10808678

Connecting via Winsock to STN

Welcome to STN International! Enter x:x

LOGINID:ssptansc1625

PASSWORD:
TERMINAL (ENTER 1, 2, 3, OR ?):2

* * * * * * * * * * Welcome to STN International * * * * * * * * * *

NEWS 1		Web Page for STN Seminar Schedule - N. America
NEWS 2	MAR 15	WPIDS/WPIX enhanced with new FRAGHITSTR display format
NEWS 3	MAR 16	CASREACT coverage extended
NEWS 4	MAR 20	MARPAT now updated daily
NEWS 5	MAR 22	LWPI reloaded
NEWS 6	MAR 30	RDISCLOSURE reloaded with enhancements
NEWS 7	APR 02	JICST-EPLUS removed from database clusters and STN
NEWS 8	APR 30	GENBANK reloaded and enhanced with Genome Project ID field
NEWS 9	APR 30	CHEMCATS enhanced with 1.2 million new records
NEWS 10	APR 30	CA/CAplus enhanced with 1870-1889 U.S. patent records
NEWS 11	APR 30	INPADOC replaced by INPADOCDB on STN
NEWS 12	MAY 01	New CAS web site launched
NEWS 13	MAY 08	CA/CAplus Indian patent publication number format defined
NEWS 14	MAY 14	RDISCLOSURE on STN Easy enhanced with new search and display fields
NEWS 15	MAY 21	BIOSIS reloaded and enhanced with archival data
NEWS 16	MAY 21	TOXCENTER enhanced with BIOSIS reload
NEWS 17	MAY 21	CA/CAplus enhanced with additional kind codes for German patents
NEWS 18	MAY 22	CA/CAplus enhanced with IPC reclassification in Japanese patents
NEWS 19	JUN 27	CA/CAplus enhanced with pre-1967 CAS Registry Numbers
NEWS 20	JUN 29	STN Viewer now available
NEWS 21	JUN 29	STN Express, Version 8.2, now available
NEWS 22	JUL 02	LEMBASE coverage updated
NEWS 23	JUL 02	LMEDLINE coverage updated
NEWS 24	JUL 02	SCISEARCH enhanced with complete author names
NEWS 25	JUL 02	CHEMCATS accession numbers revised
NEWS 26	JUL 02	CA/CAplus enhanced with utility model patents from China

NEWS EXPRESS 29 JUNE 2007: CURRENT WINDOWS VERSION IS V8.2,
CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP),
AND CURRENT DISCOVER FILE IS DATED 05 JULY 2007.

NEWS HOURS	STN Operating Hours Plus Help Desk Availability
NEWS LOGIN	Welcome Banner and News Items
NEWS IPC8	For general information regarding STN implementation of IPC 8

Enter NEWS followed by the item number or name to see news on that specific topic.

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of commercial gateways or other similar uses is prohibited and may result in loss of user privileges and other penalties.

* * * * * * * * * * * * * * * * STN Columbus * * * * * * * * * * * * * * * *

FILE 'HOME' ENTERED AT 13:28:19 ON 11 JUL 2007

=> fil caplus
COST IN U.S. DOLLARS

	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	0.21	0.21

FILE 'CAPLUS' ENTERED AT 13:28:32 ON 11 JUL 2007
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FILE COVERS 1907 - 11 Jul 2007 VOL 147 ISS 3
FILE LAST UPDATED: 10 Jul 2007 (20070710/ED)

Effective October 17, 2005, revised CAS Information Use Policies apply. They are available for your review at:

http://www.cas.org/infopolicy.html

=> e us-2004-808678/apps

E1	1		UA99-95363/PRN
E2	1		UA99-98052573/PRN
E3	0	-->	US-2004-808678/AP
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E8	1		US00-10197/AP
E9	3		US00-10220/AP
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E12	1		US00-103430/AP

=> e us2004-808678/apps

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E9	1		US2004-808682/AP

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E12             2     US2004-808689/PRN

=> s e3
L1              1 US2004-808678/AP

=> sel rn l1
E1 THROUGH E66 ASSIGNED

=> fil reg
COST IN U.S. DOLLARS                                SINCE FILE      TOTAL
                                                       ENTRY    SESSION
FULL ESTIMATED COST                                     3.50       3.71

FILE 'REGISTRY' ENTERED AT 13:30:28 ON 11 JUL 2007
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
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Property values tagged with IC are from the ZIC/VINITI data file
provided by InfoChem.

STRUCTURE FILE UPDATES:   10 JUL 2007  HIGHEST RN 942116-98-5
DICTIONARY FILE UPDATES:  10 JUL 2007  HIGHEST RN 942116-98-5

New CAS Information Use Policies, enter HELP USAGETERMS for details.

TSCA INFORMATION NOW CURRENT THROUGH December 2, 2006

  Please note that search-term pricing does apply when
  conducting SmartSELECT searches.

REGISTRY includes numerically searchable data for experimental and
predicted properties as well as tags indicating availability of
experimental property data in the original document.  For information
on property searching in REGISTRY, refer to:

http://www.cas.org/support/stngen/stndoc/properties.html

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07/11/2007

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L2 66 (108980-48-9/BI OR 109-70-6/BI OR 110-91-8/BI OR 115663-23-5/BI OR 137632-03-2/BI OR 138674-26-7/BI OR 140885-79-6/BI OR 141349-86-2/BI OR 148047-34-1/BI OR 157482-36-5/BI OR 229976-66-3/BI OR 304691-31-4/BI OR 320741-25-1/BI OR 321976-78-7/BI OR 391208-93-8/BI OR 394-32-1/BI OR 428817-87-2/BI OR 487-24-1/BI OR 500-22-1/BI OR 59835-92-6/BI OR 769948-78-9/BI OR 769948-79-0/BI OR 769948-80-3/BI OR 769948-81-4/BI OR 769948-82-5/BI OR 769948-83-6/BI OR

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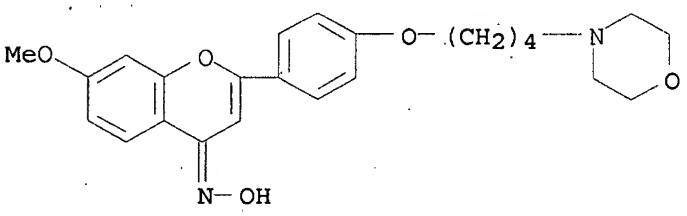
769949-13-5/BI OR 769949-14-6/BI

=> d scan 1-66
'1-66' IS NOT A VALID FORMAT FOR FILE 'REGISTRY'

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[4-(4-morpholinyl)butoxy]phenyl]-, oxime (9CI)
MF C24 H28 N2 O5
CI COM



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

The following are valid formats:

Substance information can be displayed by requesting individual fields or predefined formats. The predefined substance formats are: (RN = CAS Registry Number)

REG - RN
SAM - Index Name, MF, and structure - no RN
FIDE - All substance data, except sequence data
IDE - FIDE, but only 50 names
SQIDE - IDE, plus sequence data
SQIDE3 - Same as SQIDE, but 3-letter amino acid codes are used
SQD - Protein sequence data, includes RN
SQD3 - Same as SQD, but 3-letter amino acid codes are used
SQN - Protein sequence name information, includes RN

CALC - Table of calculated properties
EPROP - Table of experimental properties
PROP - EPROP and CALC

Any CA File format may be combined with any substance format to obtain CA references citing the substance. The substance formats must be cited first. The CA File predefined formats are:

ABS -- Abstract
APPS -- Application and Priority Information

BIB -- CA Accession Number, plus Bibliographic Data
CAN -- CA Accession Number
CBIB -- CA Accession Number, plus Bibliographic Data (compressed)
IND -- Index Data
IPC -- International Patent Classification
PATS -- PI, SO
STD -- BIB, IPC, and NCL

IABS -- ABS, indented, with text labels
IBIB -- BIB, indented, with text labels
ISTD -- STD format, indented

OBIB -------- AN, plus Bibliographic Data (original)
OIBIB ------- OBIB, indented with text labels

SBIB -------- BIB, no citations
SIBIB ------- IBIB, no citations

The ALL format gives FIDE BIB ABS IND RE, plus sequence data when it is available.
The MAX format is the same as ALL.
The IALL format is the same as ALL with BIB ABS and IND indented, with text labels.

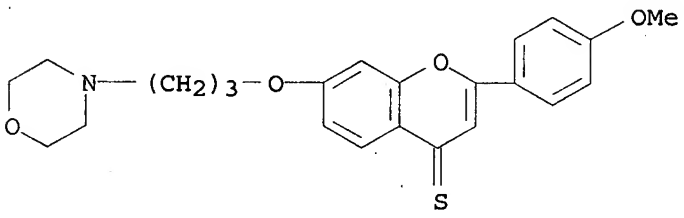
For additional information, please consult the following help messages:

HELP DFIELDS -- To see a complete list of individual display fields.
HELP FORMATS -- To see detailed descriptions of the predefined formats.
HOW MANY MORE ANSWERS DO YOU WISH TO SCAN? (1):66

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Kinase (phosphorylating), interleukin-1 receptor-associated protein, 4
MF Unspecified
CI MAN

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

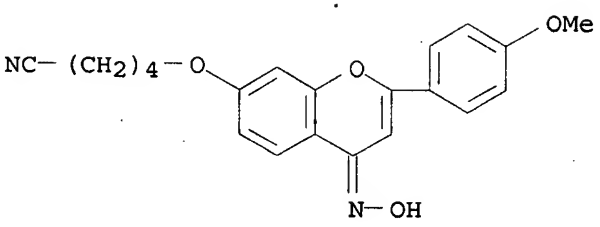
L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-thione, 2-(4-methoxyphenyl)-7-[3-(4-morpholinyl)propoxy]-(9CI)
MF C23 H25 N O4 S



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

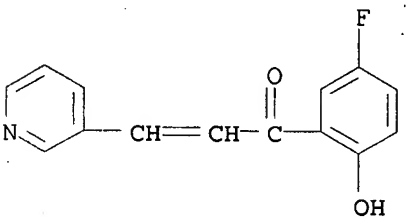
L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Pentanenitrile, 5-[[4-(hydroxyimino)-2-(4-methoxyphenyl)-4H-1-benzopyran-7-

yl]oxy]- (9CI)
MF C21 H20 N2 O4



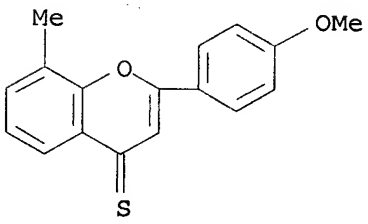
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 2-Propen-1-one, 1-(5-fluoro-2-hydroxyphenyl)-3-(3-pyridinyl)- (9CI)
MF C14 H10 F N O2



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

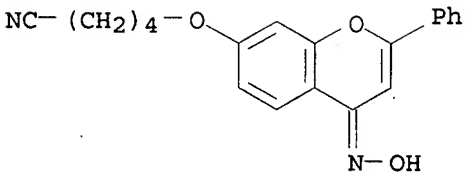
L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-thione, 2-(4-methoxyphenyl)-8-methyl- (9CI)
MF C17 H14 O2 S



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Pentanenitrile, 5-[[4-(hydroxyimino)-2-phenyl-4H-1-benzopyran-7-yl]oxy]-

(9CI)
MF C20 H18 N2 O3



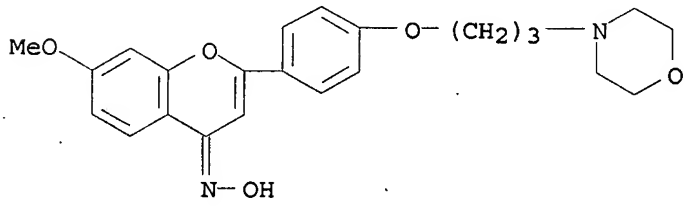
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Kinase (phosphorylating), JAK3 protein
MF Unspecified
CI MAN

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[3-(4-morpholinyl)propoxy]phenyl]-, oxime, mono(trifluoroacetate) (9CI)
MF C23 H26 N2 O5 . C2 H F3 O2

CM 1



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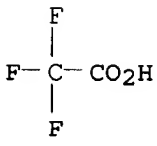
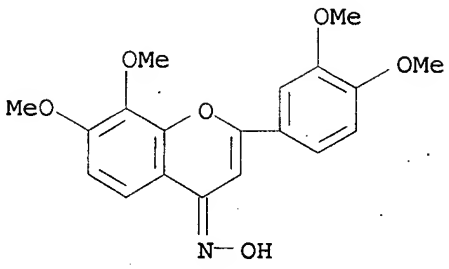
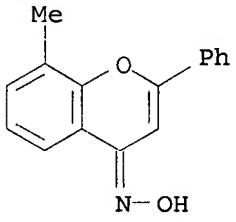


L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-7,8-dimethoxy-, oxime (9CI)
MF C19 H19 N O6



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

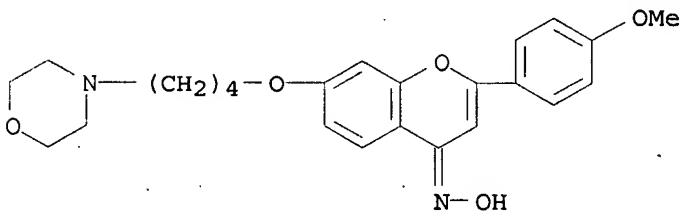
L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 8-methyl-2-phenyl-, oxime (9CI)
MF C16 H13 N O2



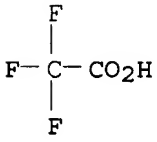
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-7-[4-(4-morpholinyl)butoxy]-, oxime, mono(trifluoroacetate) (9CI)
MF C24 H28 N2 O5 . C2 H F3 O2

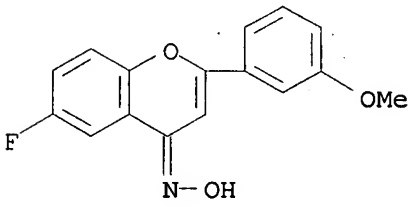
CM 1



CM 2

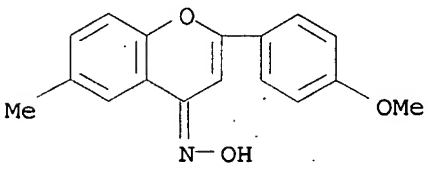


L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 6-fluoro-2-(3-methoxyphenyl)-, oxime (9CI)
MF C16 H12 F N O3



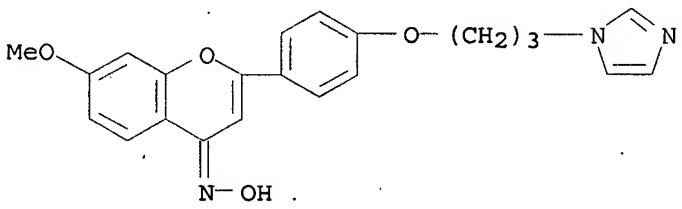
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-6-methyl-, oxime (9CI)
MF C17 H15 N O3



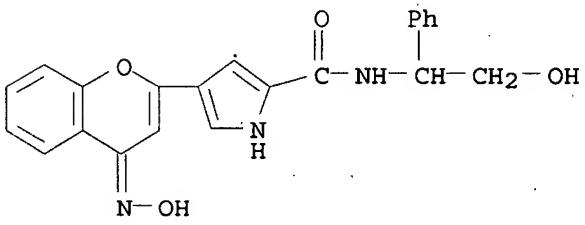
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-[4-[3-(1H-imidazol-1-yl)propoxy]phenyl]-7-methoxy-, oxime (9CI)
MF C22 H21 N3 O4



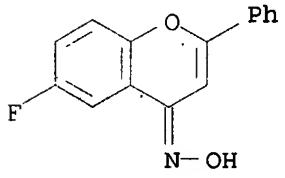
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 1H-Pyrrole-2-carboxamide, 4-[4-(hydroxyimino)-4H-1-benzopyran-2-yl]-N-(2-hydroxy-1-phenylethyl)- (9CI)
MF C22 H19 N3 O4



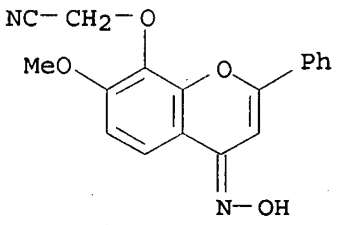
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 6-fluoro-2-phenyl-, oxime (9CI)
MF C15 H10 F N O2



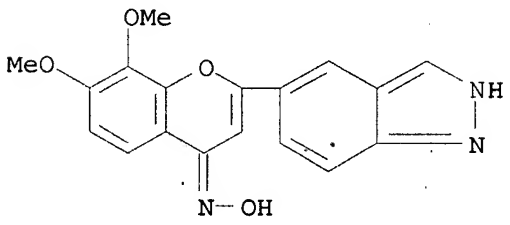
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Acetonitrile, [[4-(hydroxyimino)-7-methoxy-2-phenyl-4H-1-benzopyran-8-yl]oxy]- (9CI)
MF C18 H14 N2 O4



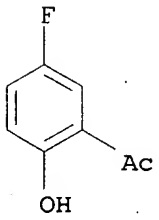
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(2H-indazol-5-yl)-7,8-dimethoxy-, oxime (9CI)
MF C18 H15 N3 O4



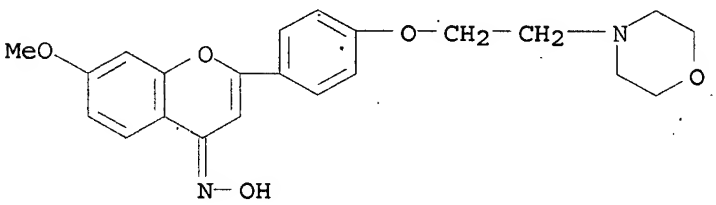
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Ethanone, 1-(5-fluoro-2-hydroxyphenyl)-
MF C8 H7 F O2
CI COM



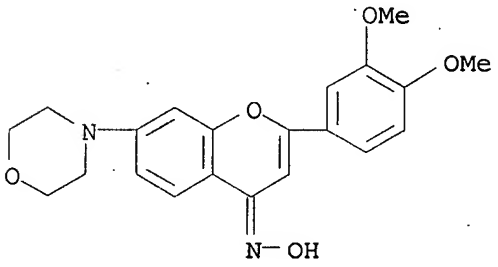
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[2-(4-morpholinyl)ethoxy]phenyl]-, oxime (9CI)
MF C22 H24 N2 O5
CI COM



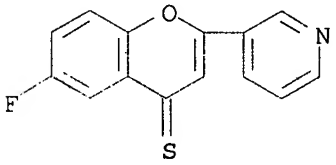
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-7-(4-morpholinyl)-, oxime (9CI)
MF C21 H22 N2 O5



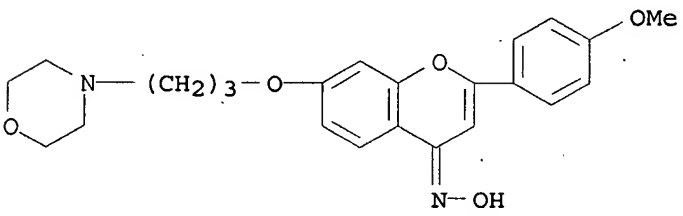
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-thione, 6-fluoro-2-(3-pyridinyl)- (9CI)
MF C14 H8 F N O S



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-7-[3-(4-morpholinyl)propoxy]-, oxime (9CI)
MF C23 H26 N2 O5
CI COM

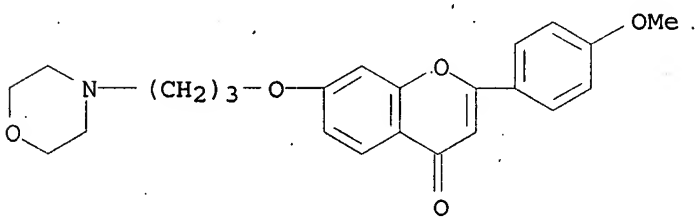


PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Kinase (phosphorylating), protein, GSK3
MF Unspecified
CI MAN

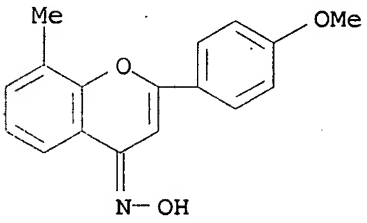
*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-7-[3-(4-morpholinyl)propoxy]-(9CI)
MF C23 H25 N O5



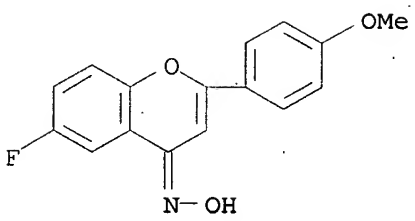
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-8-methyl-, oxime (9CI)
MF C17 H15 N O3



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

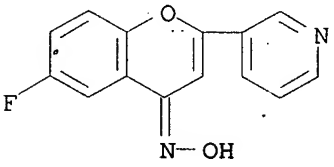
L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 6-fluoro-2-(4-methoxyphenyl)-, oxime (9CI)
MF C16 H12 F N O3



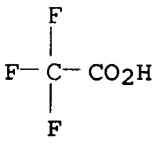
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 6-fluoro-2-(3-pyridinyl)-, oxime, mono(trifluoroacetate) (9CI)
MF C14 H9 F N2 O2 . C2 H F3 O2

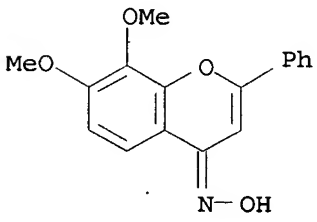
CM 1



CM 2



L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 7,8-dimethoxy-2-phenyl-, oxime (9CI)
MF C17 H15 N O4



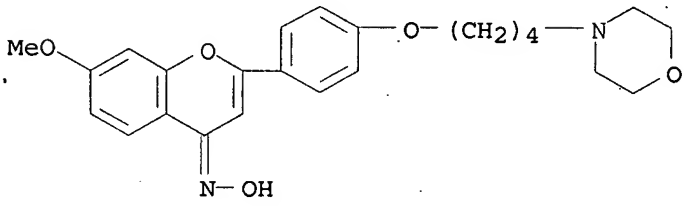
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Kinase (phosphorylating), protein ZAP-70
MF Unspecified
CI MAN

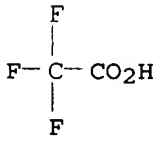
*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[4-(4-morpholinyl)butoxy]phenyl]-, oxime, mono(trifluoroacetate) (9CI)
MF C24 H28 N2 O5 . C2 H F3 O2

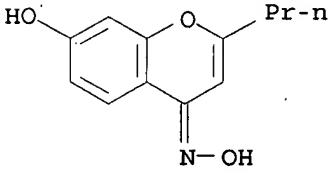
CM 1



CM 2



L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 7-hydroxy-2-propyl-, oxime (9CI)
MF C12 H13 N O3



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Kinase (phosphorylating), gene syk protein
MF Unspecified
CI MAN

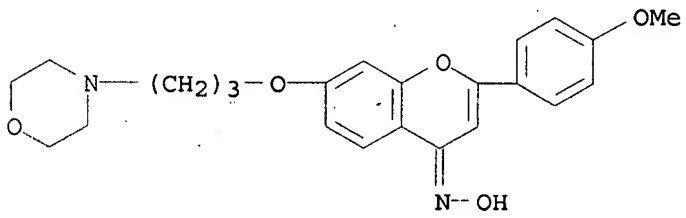
10808678

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

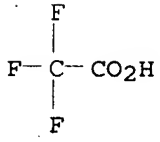
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-7-[3-(4-morpholinyl)propoxy]-, oxime, mono(trifluoroacetate) (9CI)
MF C23 H26 N2 O5 . C2 H F3 O2

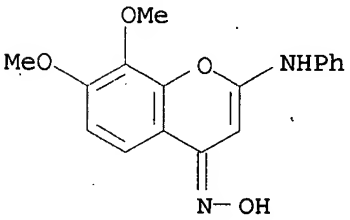
CM 1



CM 2

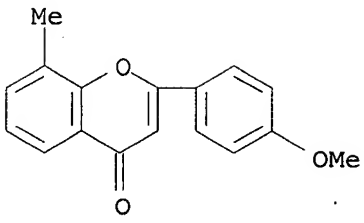


L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 7,8-dimethoxy-2-(phenylamino)-, oxime (9CI)
MF C17 H16 N2 O4



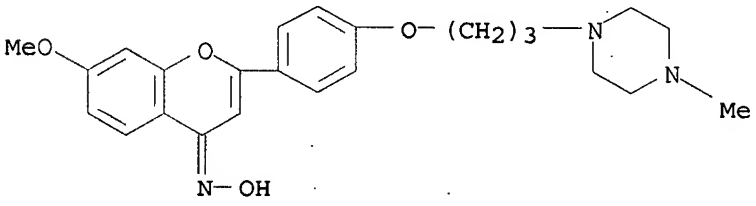
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-8-methyl- (9CI)
MF C17 H14 O3



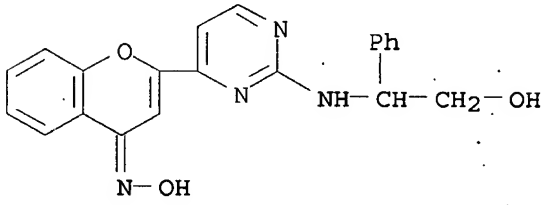
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[3-(4-methyl-1-piperazinyl)propoxy]phenyl]-, oxime (9CI)
MF C24 H29 N3 O4



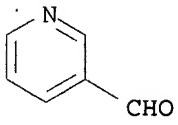
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-[2-[(2-hydroxy-1-phenylethyl)amino]-4-pyrimidinyl]-, oxime (9CI)
MF C21 H18 N4 O3



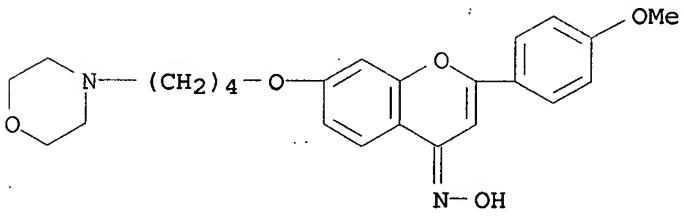
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 3-Pyridinecarboxaldehyde
MF C6 H5 N O
CI COM



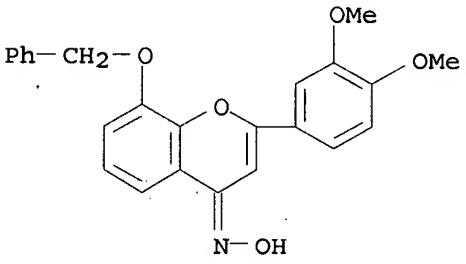
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-7-[4-(4-morpholinyl)butoxy]-, oxime (9CI)
MF C24 H28 N2 O5
CI COM



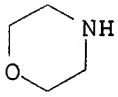
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-8-(phenylmethoxy)-, oxime (9CI)
MF C24 H21 N O5



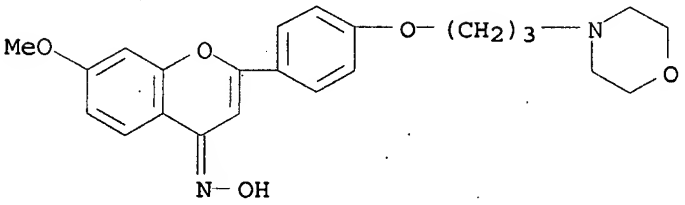
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Morpholine
MF C4 H9 N O
CI COM, RPS



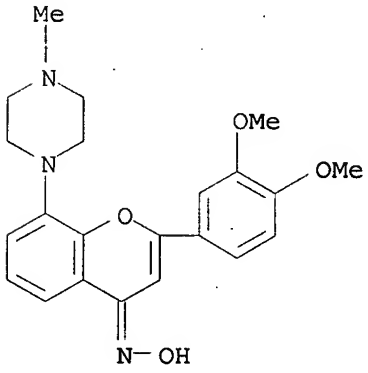
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[3-(4-morpholinyl)propoxy]phenyl]-, oxime (9CI)
MF C23 H26 N2 O5
CI COM



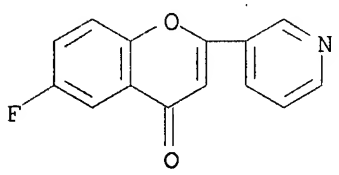
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-8-(4-methyl-1-piperazinyl)-, oxime (9CI)
MF C22 H25 N3 O4



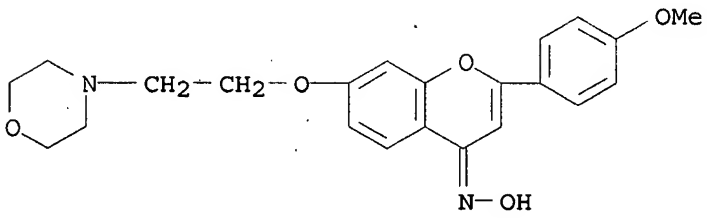
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 6-fluoro-2-(3-pyridinyl)- (9CI)
MF C14 H8 F N O2



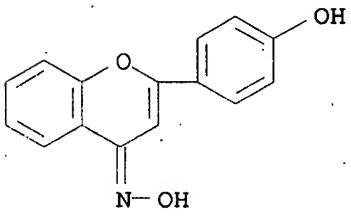
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-7-[2-(4-morpholinyl)ethoxy]-, oxime (9CI)
MF C22 H24 N2 O5
CI COM



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(4-hydroxyphenyl)-, oxime (9CI)
MF C15 H11 N O3

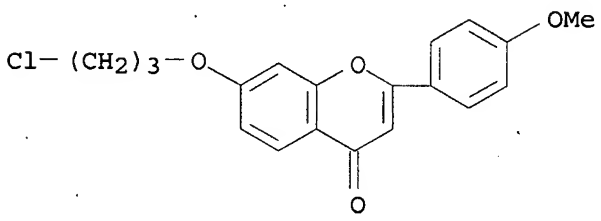


PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN

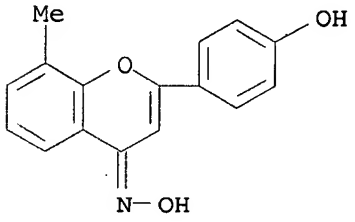
10808678

IN 4H-1-Benzopyran-4-one, 7-(3-chloropropoxy)-2-(4-methoxyphenyl)- (9CI)
MF C19 H17 Cl O4



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(4-hydroxyphenyl)-8-methyl-, oxime (9CI)
MF C16 H13 N O3



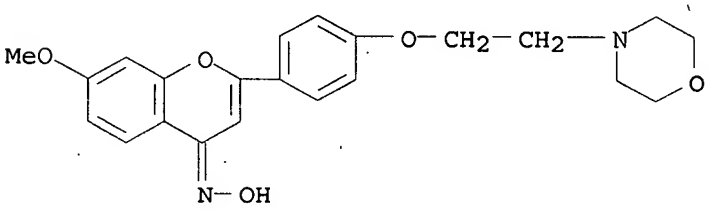
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Kinase (phosphorylating), gene Tak1 protein (9CI)
MF Unspecified
CI MAN

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

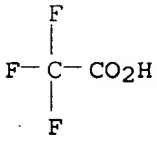
L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[2-(4-morpholinyl)ethoxy]phenyl]-, oxime, mono(trifluoroacetate) (9CI)
MF C22 H24 N2 O5 . C2 H F3 O2

CM 1

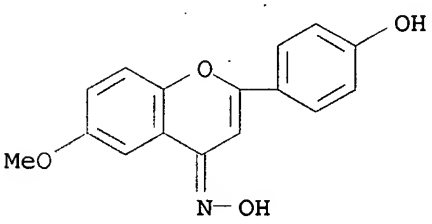


07/11/2007

CM 2



L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(4-hydroxyphenyl)-6-methoxy-, oxime (9CI)
MF C16 H13 N O4



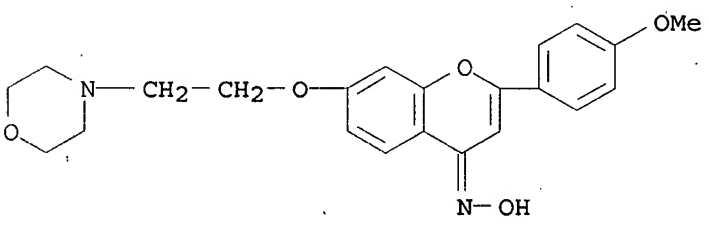
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Kinase (phosphorylating), gene cdk2 protein
MF Unspecified
CI COM, MAN

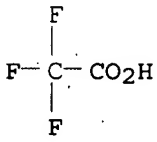
*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(4-methoxyphenyl)-7-[2-(4-morpholinyl)ethoxy]-, oxime, mono(trifluoroacetate) (9CI)
MF C22 H24 N2 O5 . C2 H F3 O2

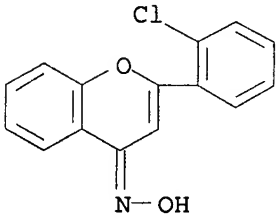
CM 1



CM 2



L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(2-chlorophenyl)-, oxime (9CI)
MF C15 H10 Cl N O2

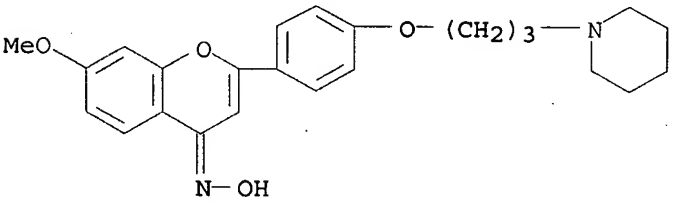


PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Kinase (phosphorylating), scatter factor receptor
MF Unspecified
CI MAN

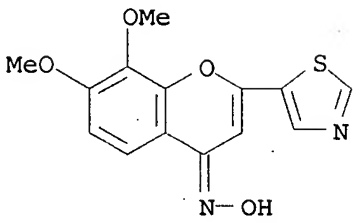
*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[3-(1-piperidinyl)propoxy]phenyl]-, oxime (9CI)
MF C24 H28 N2 O4



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 7,8-dimethoxy-2-(5-thiazolyl)-, oxime (9CI)
MF C14 H12 N2 O4 S

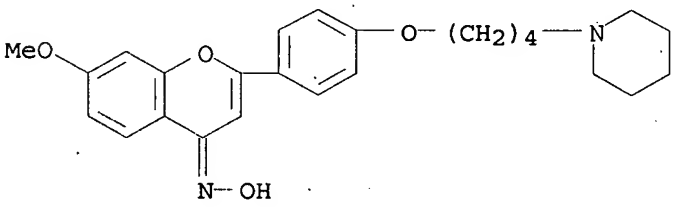


PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Kinase (phosphorylating), ribosomal protein S6, 1
MF Unspecified
CI MAN

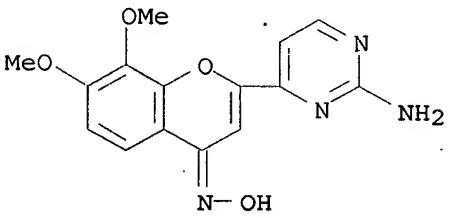
*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 7-methoxy-2-[4-[4-(1-piperidinyl)butoxy]phenyl]-, oxime (9CI)
MF C25 H30 N2 O4



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

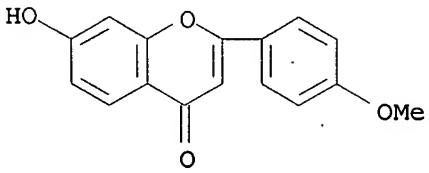
L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(2-amino-4-pyrimidinyl)-7,8-dimethoxy-, oxime (9CI)
MF C15 H14 N4 O4



10808678

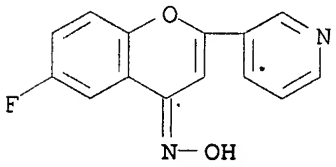
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 7-hydroxy-2-(4-methoxyphenyl)-
MF C16 H12 O4



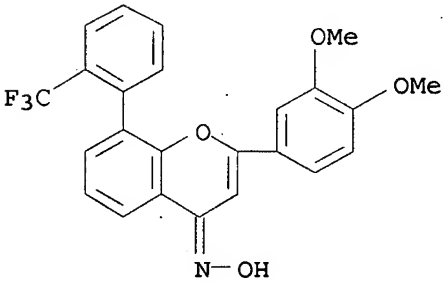
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 6-fluoro-2-(3-pyridinyl)-, oxime (9CI)
MF C14 H9 F N2 O2
CI COM



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN 4H-1-Benzopyran-4-one, 2-(3,4-dimethoxyphenyl)-8-[2-(trifluoromethyl)phenyl]-, oxime (9CI)
MF C24 H18 F3 N O4

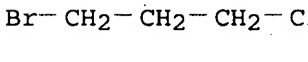


PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

07/11/2007

10808678

L2 66 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Propane, 1-bromo-3-chloro-
MF C3 H6 Br Cl
CI COM



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

ALL ANSWERS HAVE BEEN SCANNED

=> logoff y

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	1.35	5.06

STN INTERNATIONAL LOGOFF AT 13:32:19 ON 11 JUL 2007

Compound	M.Spec. $(M+H)^+$	HPLC, R_t(min). Method A or B	^{1}H-NMR
I-31	425	4.22 (A)	(500 MHz, DMSO-d_6) δ10.78 (s br, 1H), 9.98 (s, 1H), 7.89 (d, 2H), 7.79 (d, 1H), 7.08 (d, 2H), 7.03 (d, 1H), 7.00 (s, 1H), 6.89 (dd, 1H), 4.10 (t, 2H), 3.99 (d, 2H), 3.84 (s, 3H), 3.67 (t, 2H), 3.46 (d, 2H), 3.20 (m, 2H), 3.07 (m, 2H), 1.81 (m, 4H) ppm.
I-32	282	6.29 (A)	(500 MHz, DMSO-d_6) δ 10.83 (s, 1H), 7.87 (d, 2H), 7.67 (s, 1H), 7.31 (s, 2H), 7.06 (d, 2H), 6.98 (s, 1H), 3.83 (s, 3H), 2.34(s, 3H) ppm.
I-33	323	3.5 (A)	(500 MHz, DMSO-d_6) δ 10.93 (s, 1H), 7.96 (m, 2H), 7.68 (d, 1H), 7.53 (m, 3H), 7.11 (m, 2H), 5.13 (s, 2H), 3.91 (s, 3H) ppm.
I-34	423.2	2.15 (A)	(500 MHz, DMSO-d_6) δ 10.73 (s, 1H), 9.19 (s, 1H), 7.90 (d, 2H), 7.78 (d, 1H), 7.07 (d, 2H), 6.99 (s, 1H), 4.09 (t, 2H), 3.84 (s, 3H), 3.45 (d, 2H), 3.10 (m, 2H), 2.86 (q, 2H) ppm.
I-35	424.1	1.65 (A)	(500 MHz, DMSO-d_6) δ 10.65 (s, 1H), 7.88 (d, 2H), 7.77 (d, 1H), 7.06 (d, 2H), 6.98 (s, 1H), 6.88 (dd, 1H), 4.13 (t, 2H), 3.84 (s, 3H), 3.1 (m, 10H), 2.78 (s, 3H), 2.04 (m, 2H) ppm.
I-36	392.1	1.94 (A)	(500 MHz, DMSO-d_6) δ 10.70 (br s, 1H), 9.17(s, 1H), 7.87 (d, 2H), 7.83 (s, 1H), 7.78 (d, 2H), 7.71 (s, 1H), 7.02 (d, 2H), 6.99 (m, 2H), 6.88 (m, 1H), 4.41 (t, 3H) ppm, 4.12 (t, 2H), 3.85 (s, 3H), 2.33 (m, 2H) ppm.
I-37	409.2	2.05 (A)	(500 MHz, DMSO-d_6) δ 10.72 (br s, 1H), 9.42(s, 1H), 7.90 (d, 2H), 7.79 (d, 2H), 7.07 (d, 2H), 7.00 (m, 2H), 6.89 (dd, 1H), 4.15 (t, 2H), 3.86 (s, 3H), 3.51 (d, 2H) ppm, 3.22 (m, 2H), 2.92 (q, 2H), 2.16 (m, 2H), 1.83 (m, 2H), 1.67 (m, 3H), 1.39 (m, 1H) ppm.

[00221] Example 10: CDK-2 Inhibition Assay

[00222] Compounds were screened in the following manner for their ability to inhibit CDK-2 using a standard coupled enzyme assay (Fox et al., *Protein Sci.* **1998**, *7*, 2249).

[00223] To an assay stock buffer solution containing 0.1M HEPES 7.5, 10 mM $MgCl_2$, 1 mM DTT, 25 mM NaCl, 2.5 mM phosphoenolpyruvate, 300 mM NADH, 30 mg/ml pyruvate kinase, 10 mg/ml lactate dehydrogenase, 100 mM ATP, and 100 μM peptide (American

Peptide, Sunnyvale, CA) was added a DMSO solution of a compound of the present invention to a final concentration of 30 μM. The resulting mixture was incubated at 30 °C for 10 min.

[00224] The reaction was initiated by the addition of 10 μl of CDK-2/Cyclin A stock solution to give a final concentration of 25 nM in the assay. The rates of reaction were obtained by monitoring absorbance at 340 nm over a 5-minute read time at 30 °C using a BioRad Ultramark plate reader (Hercules, CA). The K_i values were determined from the rate data as a function of inhibitor concentration.

[00225] The compound numbers correspond to the compound numbers in Table 1 and were found to inhibit CDK-2. Certain compounds described herein were shown to have K_is less than 1.0 micromolar (μM).

[00226]

[00227] Example 11: cMET Inhibition Assay

[00228] Compounds were screened for their ability to inhibit cMet kinase activity using a standard coupled enzyme system (Fox et al., *Protein Sci.* **1998**, *7*, 2249). Reactions were carried out in a solution containing 100 mM HEPES (pH 7.5), 10 mM $MgCl_2$, 25 mM NaCl, 300 μM NADH, 1 mM DTT, and 1.5% DMSO. Final substrate concentrations in the assay were 200 μM ATP (Sigma Chemicals, St Louis, MO) and 10 μM polyGluTyr (Sigma Chemical Company, St. Louis). Reactions were carried out at 30 °C and 80 nM cMet. Final concentrations of the components of the coupled enzyme system were 2.5 mM phosphoenolpyruvate, 300 μM NADH, 30 μg/ml pyruvate kinase and 10 μg/ml lactate dehydrogenase.

[00229] An assay stock buffer solution was prepared containing all of the reagents listed above with the exception of ATP and a test compound of the present invention. The assay stock buffer solution (175 μl) was incubated in a 96 well plate with 5 μl of the test compound of the present invention at final concentrations spanning 0.006 μM to 12.5 μM at 30 °C for 10 min. Typically, a 12 point titration was conducted by preparing serial dilutions (from 10 mM compound stocks) with DMSO of the test compounds of the present invention in daughter plates. The reaction was initiated by the addition of 20 μl of ATP (final concentration 200 μM). Rates of reaction were obtained using a Molecular Devices Spectramax plate reader (Sunnyvale, CA) over 10 min at 30 °C. The K_i values were determined from the rate data as a function of inhibitor concentration.

[00230] The compound numbers correspond to the compound numbers in Table 1 and were found to inhibit cMET. Certain compounds described herein were shown to have K_is less than 1.0 micromolar (μM).

[00231] Example 12: Inhibition of GSK-3:

[00232] Compounds were screened for their ability to inhibit GSK-3β (AA 1-420) activity using a standard coupled enzyme system (Fox et al., *Protein Sci.* **1998**, *7*, 2249). Reactions were carried out in a solution containing 100 mM HEPES (pH 7.5), 10 mM $MgCl_2$, 25 mM NaCl, 300 μM NADH, 1 mM DTT and 1.5% DMSO. Final substrate concentrations in the assay were 20 μM ATP (Sigma Chemicals, St Louis, MO) and 300 μM peptide (American Peptide, Sunnyvale, CA). Reactions were carried out at 30 °C and 20 nM GSK-3β. Final concentrations of the components of the coupled enzyme system were 2.5 mM phosphoenolpyruvate, 300 μM NADH, 30 μg/ml pyruvate kinase and 10 μg/ml lactate dehydrogenase.

[00233] An assay stock buffer solution was prepared containing all of the reagents listed above with the exception of ATP and the test compound of interest. The assay stock buffer solution (175 μl) was incubated in a 96 well plate with 5 μl of the test compound of interest at final concentrations spanning 0.002 μM to 30 μM at 30°C for 10 min. Typically, a 12 point titration was conducted by preparing serial dilutions (from 10 mM compound stocks) with DMSO of the test compounds in daughter plates. The reaction was initiated by the addition of 20 μl of ATP (final concentration 20 μM). Rates of reaction were obtained using a Molecular Devices Spectramax plate reader (Sunnyvale, CA) over 10 min at 30°C. The K_i values were determined from the rate data as a function of inhibitor concentration.

[00234] The compound numbers correspond to the compound numbers in Table 1 and were found to inhibit GSK-3. Certain compounds described herein were shown to have K_is less than 1.0 micromolar (μM).

[00235] Example 13: SYK Inhibition Assay:

[00236] Compounds were screened for their ability to inhibit SYK using a standard coupled enzyme assay (Fox et al., *Protein Sci.* **1998**, 7, 2249). Reactions were carried out in 100 mM HEPES (pH 7.5), 10 mM $MgCl_2$, 25 mM NaCl, 1 mM DTT and 1.5% DMSO. Final substrate concentrations in the assay were 200 μM ATP (Sigma chemical Co.) and 4 μM poly Gly-Tyr peptide (Sigma Chemical Co.). Assays were carried out at 30 °C and 200 nM SYK.

Final concentrations of the components of the coupled enzyme system were 2.5 mM phosphoenolpyruvate, 300 μM NADH, 30 μg/ml pyruvate kinase and 10 μg/ml lactate dehydrogenase.

[00237] An assay stock buffer solution was prepared containing all of the reagents listed above, with the exception of SYK, DTT, and the test compound of interest of the present invention. 56 μl of the test reaction was placed in a 96 well plate followed by the addition of 1 μl of 2 mM DMSO stock containing the test compound of the present invention (final compound concentration 30 μM). The plate was pre-incubated for ~10 minutes at 30 °C and the reaction initiated by the addition of 10 μl of enzyme (final concentration 25 nM). Rates of reaction were obtained using a BioRad Ultramark plate reader (Hercules, CA) over a 5 minute read time at 30°C, and K_i values for the compounds of the present invention were determined according to standard methods.

[00238] The compound numbers correspond to the compound numbers in Table 1 and were found to inhibit SYK. Certain compounds described herein were shown to have K_is less than 1.0 micromolar (μM).

[00239] Example 14: ZAP-70 Inhibition Assay

[00240] Compounds were screened for their ability to inhibit ZAP-70 using a standard coupled enzyme assay (Fox et al., *Protein Sci.* **1998**, *7*, 2249). Assays were carried out in a mixture of 100 mM HEPES (pH 7.5), 10 mM $MgCl_2$, 25 mM NaCl , 2 mM DTT and 3% DMSO. Final substrate concentrations in the assay were 100 μM ATP (Sigma Chemicals) and 20 μM peptide (poly-4EY, Sigma Chemicals). Assays were carried out at 30 °C and 60 nM ZAP-70. Final concentrations of the components of the coupled enzyme system were 2.5 mM phosphoenolpyruvate, 300 μM NADH, 30 μg/ml pyruvate kinase and 10 μg/ml lactate dehydrogenase.

[00241] An assay stock buffer solution was prepared containing all of the reagents listed above, with the exception of ZAP-70 and the test compound of interest of the present invention. 55 μl of the stock solution was placed in a 96 well plate followed by addition of 2 μl of DMSO stock containing serial dilutions of the test compound of the present invention (typically starting from a final concentration of 15μM). The plate was preincubated for 10 minutes at 30°C and the reaction initiated by addition of 10 μl of enzyme (final concentration 60 nM). Initial reaction rates were determined with a Molecular Devices SpectraMax Plus

plate reader over a 15 minute time course. K_i data was calculated from non-linear regression analysis using the Prism software package (GraphPad Prism version 3.0a for Macintosh, GraphPad Software, San Diego California, USA).

[00242] The compound numbers correspond to the compound numbers in Table 1 and were found to inhibit ZAP-70. Certain compounds described herein were shown to have K_is less than 1.0 micromolar (μM).

[00243] Example 15: FLT-3 Inhibition Assay

[00244] Compounds were screened for their ability to inhibit FLT-3 activity using a radiometric filter-binding assay. This assay monitors the ^{33}P incorporation into a substrate poly(Glu, Tyr) 4:1 (pE4Y). Reactions were carried out in a solution containing 100 mM HEPES (pH 7.5), 10 mM $MgCl_2$, 25 mM NaCl, 1 mM DTT, 0.01% BSA and 2.5% DMSO. Final substrate concentrations in the assay were 90 μM ATP and 0.5mg/ml pE4Y (both from Sigma Chemicals, St Louis, MO). The final concentration of a compound of the present invention is generally between 0.01 and 5 μM. Typically, a 12-point titration was conducted by preparing serial dilutions from 10 mM DMSO stock of test compound. Reactions were carried out at room temperature.

[00245] Two assay solutions were prepared. Solution 1 contains 100 mM HEPES (pH 7.5), 10 mM $MgCl_2$, 25 mM NaCl, 1 mg/ml pE4Y and 180 μM ATP(containing 0.3μCi of [γ-^{33}P]ATP for each reaction). Solution 2 contains 100 mM HEPES (pH 7.5), 10 mM $MgCl_2$, 25 mM NaCl, 2 mM DTT, 0.02% BSA and 3 nM FLT-3. The assay was run on a 96 well plate by mixing 50μl each of Solution 1 and 2.5 ml of the compounds of the present invention. The reaction was initiated with Solution 2. After incubation for 20 minutes at room temperature, the reaction was stopped with 50μl of 20% TCA containing 0.4mM of ATP. All of the reaction volume was then transferred to a filter plate and washed with 5% TCA by a Harvester 9600 from TOMTEC (Hamden, CT). The amount of ^{33}P incorporation into pE4y was analyzed by a Packard Top Count Microplate Scintillation Counter (Meriden, CT). The data was fitted using Prism software to get an IC_{50} or K_i.

[00246] The compound numbers correspond to the compound numbers in Table 1 and were found to inhibit FLT-3. Certain compounds described herein were shown to have K_is less than 1.0 micromolar (μM).

[00247] Example 16: JAK-3 Inhibition Assay

[00248] Compounds of the present invention were screened for their ability to inhibit JAK activity using the method described by G. R. Brown et al., *Bioorg. Med. Chem. Lett.* **2000,** *10*, 575-579 in the following manner. Into Maxisorb plates, previously coated at 4°C with Poly (Glu, Ala, Tyr) 6:3:1 then washed with phosphate buffered saline 0.05% and Tween (PBST), was added 2 μM ATP, 5 mM $MgCl_2$, and a solution of a compound of the present invention in DMSO. The reaction was started with JAK enzyme and the plates incubated for 60 minutes at 30°C. The plates were then washed with PBST, 100 μl HRP-Conjugated 4G10 antibody was added, and the plate incubated for 90 minutes at 30°C. The plate was again washed with PBST, 100 μl TMB solution was added, and the plates were incubated for another 30 minutes at 30°C. Sulfuric acid (100 μl of a 1M solution) was added to stop the reaction and the plate was read at 450 nm to obtain the optical densities for analysis to determine IC_{50} values and K_i values.

[00249] The compound numbers correspond to the compound numbers in Table 1 and were found to inhibit JAK-3. Certain compounds described herein were shown to have K_is less than 1.0 micromolar (μM).

[00250] Example 17: p70S6K Inhibition Assay

[00251] Compounds were screened for their ability to inhibit p70S6K using a radioactive-phosphate incorporation assay at Upstate Biotechnology (Pitt and Lee, *J. Biomol. Screen.* **1996,** *1*, 47). Assays were carried out in a mixture of 8mM MOPS (pH 7.0), 10mM magnesium acetate, 0.2mM EDTA. Final substrate concentrations in the assay were 15μM ATP (Sigma Chemicals) and 100μM peptide (Upstate Ltd., Dundee, UK). Assays were carried out at 30°C and in the presence of p70S6K (5-10mU, Upstate Ltd., Dundee, UK) and [γ-^{33}P] ATP (Specific activity approx. 500 cpm/pmol, Amersham Pharmacia Biotech, Amersham, UK). An assay stock buffer solution was prepared containing all of the reagents listed above, with the exception of ATP, and the test compound of the present invention. 15 μl of the stock solution was placed in a 96 well plate followed by addition of 1μl of 40μM or 8μM DMSO stock containing the test compound of the present invention, in duplicate (final compound concentration 2μM or 0.4μM, respectively, final DMSO concentration 5%). The plate was preincubated for about 10 minutes at 30°C and the reaction initiated by addition of 4μl ATP (final concentration 15μM).

[00252] The reaction was stopped after 10 minutes by the addition of 5µl 3% phosphoric acid solution. A phosphocellulose 96 well plate (Millipore, Cat No. MAPHNOB50) was pretreated with 100µl 100mM phosphoric acid, 0.01% Tween-20 prior to the addition of the reaction mixture (20µl). The spots were left to soak for at least 5 minutes, prior to wash steps (4 × 200µl 100mM phosphoric acid, 0.01% Tween-20). After drying, 20µl Optiphase 'SuperMix' liquid scintillation cocktail (Perkin Elmer) was added to the well prior to scintillation counting (1450 Microbeta Liquid Scintillation Counter, Wallac).

[00253] Percentage inhibition of compounds of the present invention at 2µM and 0.4µM was calculated by comparing p70S6K activity with standard wells containing the assay mixture and DMSO without test compound. Compounds of the present invention showing high inhibition versus standard wells were titrated to determine IC_{50} values.

[00254] The compound numbers correspond to the compound numbers in Table 1 and were found to inhibit p70S6K. Certain compounds described herein were shown to have K_is less than 1.0 micromolar (µM).

[00255] Example 18: TAK-1 Inhibition Assay

[00256] Compounds were screened for their ability to inhibit TAK1A kinase activity using a radiometric filter binding assay. Reactions were carried out in a solution containing Buffer A (100 mM HEPES (pH 7.5), 10 mM $MgCl_2$), 25 mM NaCl, 2 mM DTT, and 1.5% DMSO. Final substrate concentrations in the assay were 50 µM ATP (a mixture of unlabeled ATP (Sigma Chemicals, St Louis, MO) and ^{33}P-labeled ATP (PerkinElmer Life Sciences, Boston, MA) for a final specific activity of 50 Ci/mol), and 12 µM bovine myelin basic protein (MBP, Vertex Pharmaceuticals, Cambridge, MA). Reactions were carried out at ambient temperature (~ 20 °C) using 20 nM TAK1A-TAB fusion protein. Under these conditions the extent of reaction is linear with time for a period of 2 hours.

[00257] A test compound of the present invention (1 µL in DMSO) was combined with ATP and Buffer A in a final volume of 47 µL in a 96 well plate. Typically, a 6 point titration was conducted by preparing serial dilutions (from 10 mM compound stocks) with DMSO of the test compounds of the present invention in daughter plates, for final concentrations spanning 0.046 µM to 3.73 µM. The reaction was initiated by the addition of 20 µl of an enzyme stock solution consisting of TAK1A-TAB fusion (described by Sugita, T. et al. in *Biochem. Biophys. Res. Comm.* **2002**, *297*, 1277-1281), MBP, Buffer A, NaCl, and DTT.

The reaction was allowed to proceed for two hours at ambient temperature, then quenched with an equal volume of 10 mM unlabeled ATP in 10% trichloroacetic acid. A 110 µL aliquot of the quenched reaction was transferred to a Multiscreen PH filter plate (Millipore, Billerica, MA) and allowed to incubate at ambient temperature overnight (typically 16-20 hours). Following incubation the filter plates were washed with 3 × 150 µL aliquots of 5% trichloroacetic acid using a modified Biotek Elx405 plate washer. A 70 µL aliquot of Microscint 20 scintillation fluid (PerkinElmer) was added to each well, and the plate was then sealed and read on a TopCount NXT microplate scintillation counter (PerkinElmer). The K_i values were determined from the rate data as a function of inhibitor concentration.

[00258] The compound numbers correspond to the compound numbers in Table 1 and were found to inhibit TAK-1. Certain compounds described herein were shown to have K_is less than 1.0 micromolar (µM).

[00259] Example 19: IRAK-4 Inhibition Assay

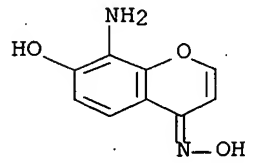
[00260] Compounds were screened for their ability to inhibit IRAK-4 using a standard coupled enzyme assay (Fox *et al., Protein Sci.* **1998** *7*, 2249). Assays were carried out in a solution containing 100 mM HEPES (pH 7.5), 10 mM MgCl2, 25 mM NaCl, 2 mM DTT, and 2.5% DMSO. Final substrate concentrations in the assay were 600 µM ATP (Sigma Chemicals, St Louis, MO) and 300 µM custom peptide substrate (HMRSAMSGLHLVKRR (American Peptide, Sunnyvale, CA)). Final enzyme concentration in the assay was 30 nM IRAK-4. Final concentrations of the coupled enzyme system components were 2.5 mM phosphoenolpyruvate, 300 µM NADH, 30 µg/ml pyruvate kinase and 10 µg/ml lactate dehydrogenase. Assays were carried out at 30 °C.

[00261] Two assay solutions were prepared. Solution 1 contains 100 mM HEPES (pH 7.5), 10 mM MgCl2, 28 mM NaCl, 2.8 mM phosphoenolpyruvate, 335 µM NADH, 335 µM peptide, and 670 µM ATP. Solution 2 contains 100 mM HEPES (pH 7.5), 10 mM MgCl2, 335 µg/ml pyruvate kinase, 112 µg/ml lactate dehydrogenase, 22 mM DTT, and 335 nM IRAK-4. 60 µl of the Solution 1 was placed in a 384 well plate, and the plate was preincubated for about 15 minutes at 30°C. The reaction was initiated by addition of 1 µl of solution containing 667µM of the compound of the present invention dissolved in DMSO (final compound concentration 10 µM) and 6 µl of Solution 2. Rates of reaction were obtained by monitoring the change in absorbance at 340nm over a 6 minute read time at 30°C

using a Molecular Devices SpectraMax Plus plate reader. Compounds showing greater than 50% inhibition were selected for further testing. These selected compounds were assayed again using serial dilutions prepared from the10mM DMSO stock vial. The concentration of these titrations typically ranged from 3 nM to 30 μM. The data was fit using Prism software to obtain an IC_{50}.

[00262] The compound numbers correspond to the compound numbers in Table 1 and were found to inhibit IRAK-4. Certain compounds described herein were shown to have K_is less than 1.0 micromolar (μM).

RN 108128-33-2 CAPLUS
CN Chromone, 8-amino-7-hydroxy-, oxime (6CI) (CA INDEX NAME)

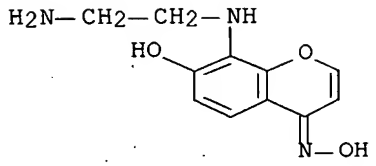


L9 ANSWER 41 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1960:128789 CAPLUS Full-text
DOCUMENT NUMBER: 54:128789
ORIGINAL REFERENCE NO.: 54:24624b-d
TITLE: Reactions of chlorinated furanidines. II. Synthesis of substituted 2-alkoxy-3-chlorotetrahydrofurans
AUTHOR(S): Kratochvil, M.
CORPORATE SOURCE: Vojenska tech. akad. A. Zapotockeho, Brno, Czech.
SOURCE: Collection of Czechoslovak Chemical Communications (1960), 25, 1351-8
CODEN: CCCCAK; ISSN: 0010-0765
DOCUMENT TYPE: Journal
LANGUAGE: Russian

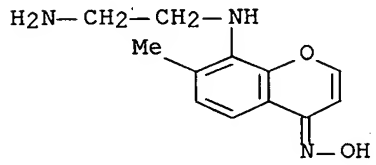
AB cf. CA 52, 16329f. Allowing to react at -5 to 0° 0.5 mole 2,3-dichlorotetrahydrofuran (I) with 0.05 mole ethylene oxide in 50 ml. dry CCl4 in the presence of 0.02-0.03 g. anhydrous ZnCl2 gives 86-8% title compds. (II) (alkyl, b.p./mm., nD20, and d20 given): CH2CH2Cl, 110.5°/15, 1.4757, 1.2816; CHMeCH2Cl, 76-6.5°/2, 1.4676, 1.2190; CH(CH2Cl)2, 121-2°/6, 1.4902, 1.3533; CH2CHClCH2Cl, 118-20°/6, 1.4906, 1.3586; CH(CH2OH)CH2Cl, 120-2°/3, 1.4882, 1.3315, CH2CHClCH2Cl, 163.5-4.0°/2, 1.4939, 1.3410; CH2CH(OH)CH2Cl, 122-4°/3, 1.4890, 1.3285; (CH2)3Cl, 110-12°/12, 1.4711, 1.2308. II (R = CH2CH:CH2) was obtained in 104-g. yield by adding at 45-50° 0.05 g. ZnCl2 and 58 g. allyl alc. to 141 g. I, refluxing the mixture 3 hrs. to 70-5°, and working up as usual to give a liquid, b14 78-9°, nD20 1.4609, d20 1.1256. Infrared spectra were charted.

IT 100128-04-9 100138-36-1 100138-65-6
100705-82-6 100706-15-8 100951-92-6
101437-16-5 101437-25-6 101867-86-1
101867-93-0 101867-94-1 101867-95-2
101889-35-4 101889-36-5 101889-40-1
101869-41-2 102371-47-1 102377-32-?
102659-19-8 102659-20-1 102659-21-2
112441-18-6 112441-19-7
(Derived from data in the 6th Collective Formula Index (1957-1961))

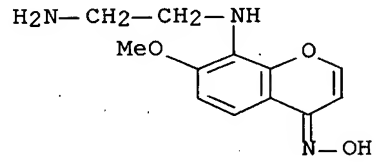
RN 100128-04-9 CAPLUS
CN Chromone, 8-[(2-aminoethyl)amino]-7-hydroxy-, oxime (6CI) (CA INDEX NAME)



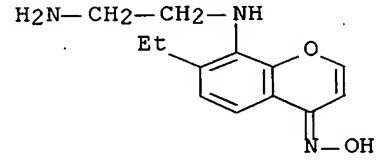
RN 100138-36-1 CAPLUS
CN Chromone, 8-[(2-aminoethyl)amino]-7-methyl-, oxime (6CI) (CA INDEX NAME)



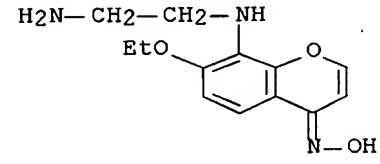
RN 100138-65-6 CAPLUS
CN Chromone, 8-[(2-aminoethyl)amino]-7-methoxy-, oxime (6CI) (CA INDEX NAME)



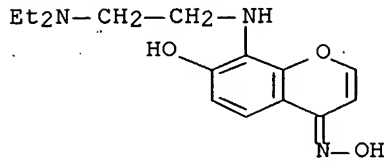
RN 100705-82-6 CAPLUS
CN Chromone, 8-[(2-aminoethyl)amino]-7-ethyl-, oxime (6CI) (CA INDEX NAME)



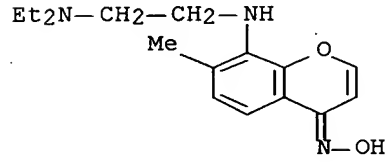
RN 100706-15-8 CAPLUS
CN Chromone, 8-[(2-aminoethyl)amino]-7-ethoxy-, oxime (6CI) (CA INDEX NAME)



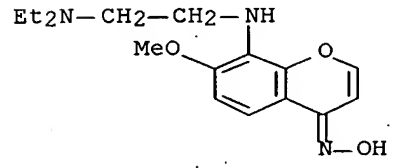
RN 100951-92-6 CAPLUS
CN Chromone, 8-[(2-diethylaminoethyl)amino]-7-hydroxy-, oxime (6CI) (CA INDEX NAME)



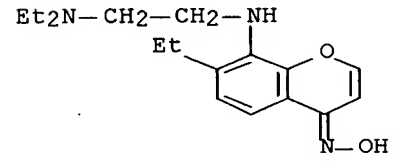
RN 101437-16-5 CAPLUS
CN Chromone, 8-[(2-diethylaminoethyl)amino]-7-methyl-, oxime (6CI) (CA INDEX NAME)



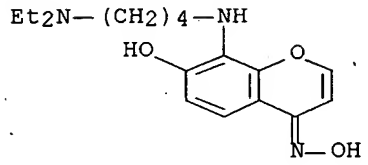
RN 101437-25-6 CAPLUS
CN Chromone, 8-[(2-diethylaminoethyl)amino]-7-methoxy-, oxime (6CI) (CA INDEX NAME)



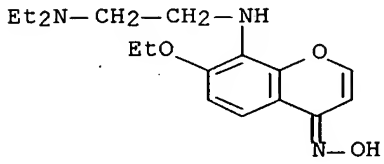
RN 101867-86-1 CAPLUS
CN Chromone, 8-[(2-diethylaminoethyl)amino]-7-ethyl-, oxime (6CI) (CA INDEX NAME)



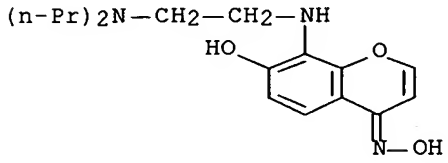
RN 101867-93-0 CAPLUS
CN Chromone, 8-[(4-diethylaminobutyl)amino]-7-hydroxy-, oxime (6CI) (CA INDEX NAME)



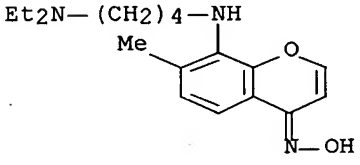
RN 101867-94-1 CAPLUS
CN Chromone, 8-[(2-diethylaminoethyl)amino]-7-ethoxy-, oxime (6CI) (CA INDEX NAME)



RN 101867-95-2 CAPLUS
CN Chromone, 8-[(2-dipropylaminoethyl)amino]-7-hydroxy-, oxime (6CI) (CA INDEX NAME)

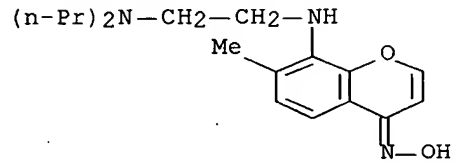


RN 101889-35-4 CAPLUS
CN Chromone, 8-[(4-diethylaminobutyl)amino]-7-methyl-, oxime (6CI) (CA INDEX NAME)

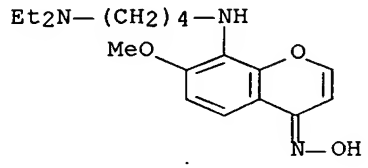


RN 101889-36-5 CAPLUS

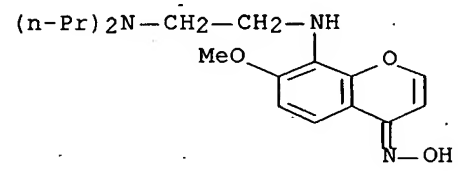
CN Chromone, 8-[(2-dipropylaminoethyl)amino]-7-methyl-, oxime (6CI) (CA INDEX NAME)



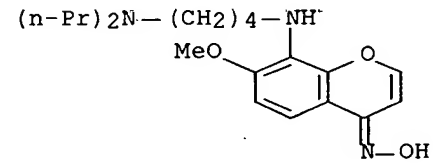
RN 101889-40-1 CAPLUS
CN Chromone, 8-[(4-diethylaminobutyl)amino]-7-methoxy-, oxime (6CI) (CA INDEX NAME)



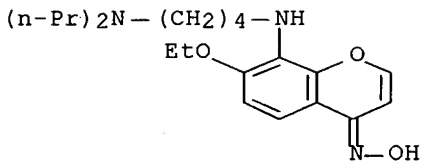
RN 101889-41-2 CAPLUS
CN Chromone, 8-[(2-dipropylaminoethyl)amino]-7-methoxy-, oxime (6CI) (CA INDEX NAME)



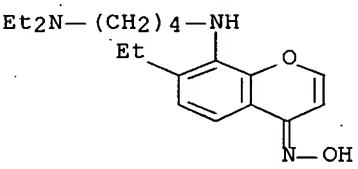
RN 102371-47-1 CAPLUS
CN Chromone, 8-[(4-di-propylaminobutyl)amino]-7-methoxy-, oxime (6CI) (CA INDEX NAME)



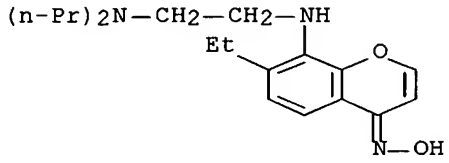
RN 102377-32-2 CAPLUS
CN Chromone, 8-[(4-dipropylaminobutyl)amino]-7-ethoxy-, oxime (6CI) (CA INDEX NAME)



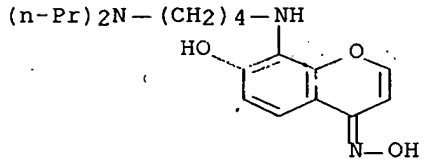
RN 102659-19-8 CAPLUS
CN Chromone, 8-[(4-diethylaminobutyl)amino]-7-ethyl-, oxime (6CI) (CA INDEX NAME)



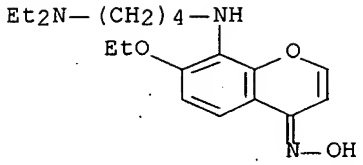
RN 102659-20-1 CAPLUS
CN Chromone, 8-[(2-dipropylaminoethyl)amino]-7-ethyl-, oxime (6CI) (CA INDEX NAME)



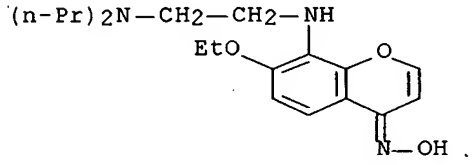
RN 102659-21-2 CAPLUS
CN Chromone, 8-[(4-dipropylaminobutyl)amino]-7-hydroxy-, oxime (6CI) (CA INDEX NAME)



RN 112441-18-6 CAPLUS
CN Chromone, 8-[(4-diethylaminobutyl)amino]-7-ethoxy-, oxime (6CI) (CA INDEX NAME)



RN 112441-19-7 CAPLUS
CN Chromone, 8-[(2-dipropylaminoethyl)amino]-7-ethoxy-, oxime (6CI) (CA INDEX NAME)

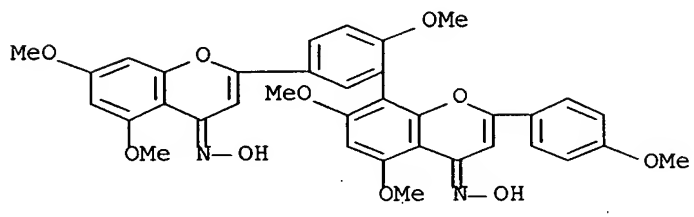


L9 ANSWER 42 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1960:86472 CAPLUS Full-text
DOCUMENT NUMBER: 54:86472
ORIGINAL REFERENCE NO.: 54:16449b-d
TITLE: Synthesis of ginkgetin tetramethyl ether
AUTHOR(S): Nakazawa, Koichi
CORPORATE SOURCE: Coll. Pharmacy, Gifu
SOURCE: Chem. & Pharm. Bull. (Tokyo) (1959), 7, 748-9
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB The synthesis was given for methylated biflavonyl (I), m. 238° (HCONMe2), which was identical with the title compound (CA 44, 9441i). 2-Acetyl-3,5-dimethoxyphenyl 3-iodoanisate and 2-acetyl-6-iodo-3,5- dimethoxyphenyl anisate were isomerized to the ketones in pyridine by KOH (yields of 40% and 78%, resp.). The ketones were cyclized to the flavones (91% and 85% yields, resp.) in H2SO4 and HOAc and the iodinated flavone (1 mole each) was converted to 28% I by heating 8 hrs. with an equal weight of Cu powder in boiling HCONMe2. I was slightly soluble in MeOH, EtOH, and dioxane, and gave a dioxime (m. 252°).

IT 107225-53-6P, Ginkgetin, tetra-O-methyl-, dioxime
RL: PREP (Preparation)
(preparation of)

RN 107225-53-6 CAPLUS
CN 3''',8-Biflavone, 4',4''',5,5'',7,7''-hexamethoxy-, dioxime (7CI) (CA INDEX NAME)

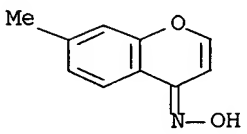


L9 ANSWER 43 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1959:122072 CAPLUS Full-text
DOCUMENT NUMBER: 53:122072
ORIGINAL REFERENCE NO.: 53:21909a-g
TITLE: 4-Chromanones. III. Alkylation and bromination of chromanones. Transformation into chromones
AUTHOR(S): Colonge, J.; Guyot, A.
CORPORATE SOURCE: Fac. sci., Lyon
SOURCE: Bulletin de la Societe Chimique de France (1958) 329-34
CODEN: BSCFAS; ISSN: 0037-8968
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

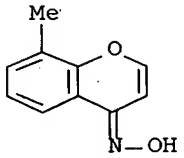
AB By reaction with tert-C5H11ONa [in C6H4(Me)2] and MeI (cf. Vavon and Conia, C.A. 41, 721a), and purification via the semicarbazones, the following compds. were obtained from, resp., I, IV, and X: [m.p., b.p. (pressure in mm.); semicarbazone m.p. given]: VII (-, 154°(40); 238°]; VIII [34, 156°(25); 233°], and IX [42.5, 185-95°(7); 271°]. By refluxing the chromanones in Et2O with 1.1 moles Br, the 3-bromo-derivs. of the following chromanones were obtained (chromanone, % yield, m.p. given): I, 78, 70°; VII, 57, 40°; IV, 77, 74°; III, 48, 89°; II, 64, 90°; VI, 60, 93°; V, 58, 153°; XI, 77, 116°; and X, 76, 113°. With 2.2 moles Br, 3,3-dibromo derivs. were obtained (as above): I, 98, 72°; IV, 94 (yield on monobromocompd.), 119°; and II, 66 (yield on monobromocompd.), 106°. The bromocompds. attacked skin and mucosas. To determine Br, the substances had to be refluxed for 7 hrs. with 0.5N KOH in glycol and the Br ion titrated potentiometrically (Lange and Berger, C.A. 25, 1179). By refluxing 48 g. 3-bromo-4-chromanone (XII) and 100 g. PhNMe2, 55% chromone (XIII) (m. 57°, oxime m. 184°) was obtained. Stirring at room temperature for 25 hrs. 20 g. XII, 13 g. HNEt2, and 50 ml. H2O yielded 72% 3-diethylamino-4-chromanone (m. 76°) which, refluxed with HCl, gave XIII, which was also obtained in 65% yield by (CO2H)2 hydrolysis of 3-(piperidino)-4-chromanone (m. 117°, obtained in 86% yield from XII and piperidine in petr. ether). From 3-bromo-6-methyl-4-chromanone, 6-methyl-3-(piperidino)-4-chromanone (m. 131°) was obtained in 65% yield. With (CO2H)2 it gave a 57% yield of 6-methylchromone (m. 88°, oxime m. 174°). Action of HNEt2 on 3-bromo-7-methyl-4-chromanone gave directly 58% 7-methylchromone (m. 73°, oxime m. 185°). From 3-bromo-8-methyl-4-chromanone, HNEt2 and (CO2H)2 hydrolysis yielded 56% 8-methylchromone (m. 84°, oxime m. 107°). Reaction of Zn powder with alc. and 2,3-dibromo-4-chromanone (XIV) [Arndt, Ber. 58, 1612(1925)] yielded XIII, which was not obtained from 3,3-dibromo-4-chromanone (XV). 3-Bromochromone (XVI) (m. 93°) could be obtained: (a) from XIV by addition of piperidine in Et2O; (b) from XV by HNEt2, HNMe2 or, best, piperidine addition Hydrogenation (Pd) of XVI yielded XII. The product, m. 65°, obtained by Arndt (loc. cit.) was not XVI (infrared spectrum). XVI with piperidine yielded 3-(piperidino)chromone (m. 124°) which, with (CO2H)2, gave XIII. Addition of 5.5 g. Br to 5 g. 6-methylchromone yielded 27% 2,3-dibromo-6-methyl-4-chromanone (m. 94°), which gave with piperidine: (a) in Et2O solution, 60% 3-bromo-6-methylchromone (m. 101°); (b) directly (with cooling), 52% 6-methyl-3-(piperidino)chromone (m. 128.5°). Similarly were obtained 2,3-dibromo-7-methyl-4-chromanone (yield 34%, m. 89°), and 3-bromo-7-methylchromone (yield 64%, m. 107°). Addition of Br to 8-methylchromone in Et2O gave a product, m. 112°, giving with HNEt2 3-bromo-8-methylchromone (m. 114°), also obtained from 3,3-dibromo-8-methyl-4-chromanone and HNEt2 (or piperidine).

IT 103261-60-5P, Chromone, 7-methyl-, oximes 103261-61-6P, Chromone, 8-methyl-, oximes 103264-01-3P, Chromone, 6-methyl-, oximes
RL: PREP (Preparation)

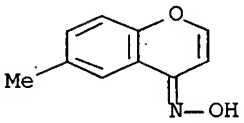
(preparation of)
RN 103261-60-5 CAPLUS
CN Chromone, 7-methyl-, oxime (6CI) (CA INDEX NAME)



RN 103261-61-6 CAPLUS
CN Chromone, 8-methyl-, oxime (6CI) (CA INDEX NAME)



RN 103264-01-3 CAPLUS
CN Chromone, 6-methyl-, oxime (6CI) (CA INDEX NAME)

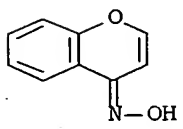


L9 ANSWER 44 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1959:122071 CAPLUS Full-text
DOCUMENT NUMBER: 53:122071
ORIGINAL REFERENCE NO.: 53:21908d-i,21909a
TITLE: 4-Chromanones. II. Cyclodehydration of 3-aryloxyalkanoic acids, tertiary chromanols and chromenes
AUTHOR(S): Colonge, J.; Guyot, A.
CORPORATE SOURCE: Fac. sci., Lyon
SOURCE: Bulletin de la Societe Chimique de France (1958) 325-8
CODEN: BSCFAS; ISSN: 0037-8968
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 53:122071
AB cf. C.A. 52, 6280h. 3-Phenoxypropionic acid (85 g.) was dissolved in 250 ml. MePh. Water (15 ml.) was added and, slowly, 50 g. P2O5. After 1 hr. refluxing with stirring, another 50 g. P2O5 was added. After 1 hr. the MePh layer was washed with Na2CO3 solution and the aqueous one diluted with H2O and extracted with Et2O. Both solns. were washed with H2O, dried (Na2SO4) and distilled to yield 82% 4-chromanone (I), m. 38.5° (oxime, m. 138°). From 3-o-

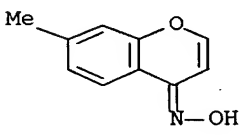
tolyloxypropionic, 3-m-tolyloxypropionic, 3-p-tolyloxypropionic, 3-o-methoxyphenyloxypropionic, 3-m-methoxyphenyloxypropionic, 3-p-methoxyphenyloxypropionic, m-phenylenebis(3-oxypropionic), p-phenylenebis(3-oxypropionic), 2-methyl-3-phenoxypropionic, 2-methyl-3-p-tolyloxypropionic, 2-methyl-3-m-tolyloxypropionic, 2-methyl-3-o-tolyloxypropionic, and 2-methyl-3-β-naphthoxypropionic acids were obtained, resp.: [% yield, m.p., b.p. (pressure in mm.); oxime m.p. given]: 8-methyl-4-chromanone (II) [81, 29.5°, 170°(42); 123°], 7-methyl-4-chromanone (III) [69, viscous oil, 151°(27), d21 1.1576, n21D 1.544; 98°], 6-methyl-4- chromanone (IV) [68, 34°, 160-2°(28); 84°], 8-methoxy-4-chromanone [25, 89°, -; 146°], 7-methoxy-4-chromanone (V) [58, 55°, -; 134°]; 6-methoxy-4-chromanone (VI) [60.5, 48°, 178-80°(23); 120°], 7-(2-carboxyethoxy)-4-chromanone [54 (in dioxane), 169°, -; 223°], 1,5-dioxa-2,3,4,6,7,8-hexahydro-4,8- anthracenedione [10(in anisole), 234° (decompose), -; dioxime decompose 300°], 3-methyl-4-chromanone (VII) [60, liquid, 154°(40), d26.5 1.131, n26.5D 1.5563; 156°], 3,6-dimethyl-4-chromanone (VIII) [66, 33°, 158-60° (30); 129°], 3,7-dimethyl-4- chromanone [53, 54°, 159-62°(30); 120°], 3,8-dimethyl-4-chromanone [58, 65°, 170-2°(40); 118°], and 3-methyl-5,6-benzo-4-chromanone (IX) [53, 42.5°, -; 110°]. By the method of Bachman and Levine (C.A. 42, 169h) 3-β-naphthoxypropionitrile and 3-α-naphthoxypropionitrile were converted into, resp., 5,6-benzo-4-chromanone (X) [82, 44°, 160-1°(2.5); 112°], and 7,8-benzo-4-chromanone (XI) [68 105°, -; 137°]. By Grignard reaction with MeMgI and hydrolysis, the chromanones were transformed into the resp. 4-methyl-4-chromanols (chromanone, % yield, m.p.): I, 70, 107°; II, 97, 72°; III, 94, 80°; IV, 94, 116°; V, 79, 61°; VI, 79, 71°; X, 95, 124°; and XI, 77, 87°. By refluxing (and separating dist. H2O) in C6H6 solution with a few dg. anhydrous CuSO4, these tertiary chromanols yielded 4-methyl-3-chromenes, separated by filtration and C6H6 evaporation Thus the following 4-methyl-3-chromenes were obtained [% yield, b.p. (pressure in mm.), dt (t in °C), ntD given]: unsubstituted [83, 102°(8), 1.074(23°), 1.590]; 6-methyl- [86, 134°(22), 1.041(24°), 1.573]; 7-methyl- [86, 123°(16), 1.054(21°), 1.574]; 8-methyl- [79, 128°(22), 1.054(20°), 1.572]; 5,6-benzo- [58, 150°(3), 1.195(17°), 1.659]; 7,8 benzo- [58, 170°(7), 1.194(19°), 1.657]; 6-methoxy- [62, 147°(6), 1.140(19°), 1.576]; and 7-methoxy- [60, 144°(6), 1.143(19°), 1.575]. 4-Ethyl-3-chromene (b10 125°, d23 1.076, nD 1.569) was prepared directly from I and EtMgI in 70% yield, as the alc. decomposed

IT 61348-46-7 103261-60-5 103261-61-6
103264-01-3
(Derived from data in the 6th Collective Formula Index (1957-1961))

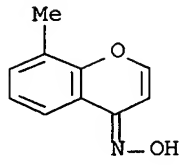
RN 61348-46-7 CAPLUS
CN 4H-1-Benzopyran-4-one, oxime (9CI) (CA INDEX NAME)



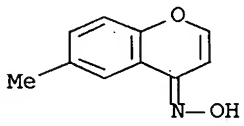
RN 103261-60-5 CAPLUS
CN Chromone, 7-methyl-, oxime (6CI) (CA INDEX NAME)



RN 103261-61-6 CAPLUS
CN Chromone, 8-methyl-, oxime (6CI) (CA INDEX NAME)

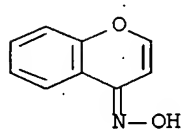


RN 103264-01-3 CAPLUS
CN Chromone, 6-methyl-, oxime (6CI) (CA INDEX NAME)

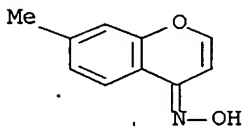


L9 ANSWER 45 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1959:122070 CAPLUS Full-text
DOCUMENT NUMBER: 53:122070
ORIGINAL REFERENCE NO.: 53:21907i,21908a-d
TITLE: Biflavonyls, a new class of natural product. The structures of ginkgetin, isoginkgetin, and sciadopitysin
AUTHOR(S): Baker, W.; Finch, A. C. M.; Ollis, W. D.; Robinson, K. W.
CORPORATE SOURCE: Univ. Bristol, UK
SOURCE: Proc. Chem. Soc. (1959) 91-2
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
GI For diagram(s), see printed CA Issue.
AB cf. Nakazawa, Yakugaku Zasshi 61, 228(1941). Ginkgetin (I) (R = H, R' = Me) (Ia) m. 342-4°, and isoginkgetin (I, R = Me, R' = H) (II), m. 355°, were isolated from Ginkgo biloba leaves. Methylation of Ia and II gave the same tetra-Me ether (III) while demethylation gave the same hexahydric phenol. Ia and II gave different tetra-acetates. Trimethylation of sciadopitysin (I, R = R' = Me) (IV) (Kariyone and Kawano, C.A. 50, 16759g) gave III. III with alkaline H2O2 gave p-anisic acid (V), 2,4,6-HO(MeO)2C6H2CO2H and C12H4(OMe)3(OH)(CO2H)2. Similarly, IV gave 4-methoxyisophthalic acid (VI) and V. Alkaline hydrolysis of IV gave C23H14O5(OMe)2 and 2,6,4-(HO)2(MeO)C6H2Ac. Infrared and ultraviolet spectra of IV placed hydroxyls in the 5''- and 7''-positions. II with alkaline H2O2 gave V and VI. Similarly, I gave VI and p-HOC6H4CO2H. Infrared and ultraviolet spectra of I placed hydroxyls at positions 5, 5'', and 7''. The 3',8''-biflavonoid structure was preferred on the bases of mechanism of biosynthesis, ease of methylation, and lack of optical activity.
IT 61348-46-7 103261-60-5 103261-61-6

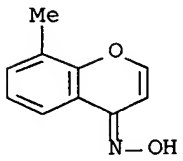
103264-01-3
(Derived from data in the 6th Collective Formula Index (1957-1961))
RN 61348-46-7 CAPLUS
CN 4H-1-Benzopyran-4-one, oxime (9CI) (CA INDEX NAME)



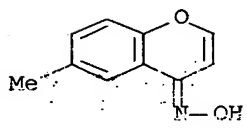
RN 103261-60-5 CAPLUS
CN Chromone, 7-methyl-, oxime (6CI) (CA INDEX NAME)



RN 103261-61-6 CAPLUS
CN Chromone, 8-methyl-, oxime (6CI) (CA INDEX NAME)



RN 103264-01-3 CAPLUS
CN Chromone, 6-methyl-, oxime (6CI) (CA INDEX NAME)



L9 ANSWER 46 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1959:17230 CAPLUS Full-text
DOCUMENT NUMBER: 53:17230
ORIGINAL REFERENCE NO.: 53:3203e-g
TITLE: Flavonoids of the leaves of Coniferae and allied plants. II. Flavonoids from the leaves of Cycas revoluta and Cryptomeria japonica var. araucarioides

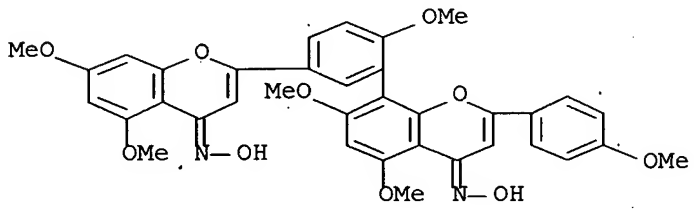
AUTHOR(S): Kariyone, Tatsuo; Sawada, Tokunosuke
SOURCE: Yakugaku Zasshi (1958), 78, 1013-15
CODEN: YKKZAJ; ISSN: 0031-6903
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB Extraction of 5 kg. dried leaves of C. revoluta in a similar way yielded 4.1 g. sotetsuflavone (I), C31H20O10.H2O, columns, m. 264-5° (decomposition) (MeOH). I gives penta-Ac derivative, needles, m. 233-4°; tri-Me derivative (II), m. 281-2° (identical with kayaflavone mono-Me ether by mixed m.p.); II diacetate, m. 228-30° (identical with mono-Me kayaflavone diacetate by mixed m.p.); penta-Me derivative (III), m. 220-1° (identical with trimethylkayaflavone and tetramethylginkgetin by mixed m.p.); III dioxime, m. 249-50° (identical with trimethylkayaflavone dioxime by mixed m.p.); penta-Et derivative, m. 234-5° (identical with tetraethylginkgetin and triethylkayaflavone by mixed m.p.). Demethylation of I and its acetylation yielded demethylkayaflavone hexaacetate, m. 239-40°. Thus, I is monodemethylginkgetin. Extraction of dried leaves of C. japonica var. araucarioides in a similar way gives kayaflavone, m. 314-5° (decomposition), sciadopitysin, m. 286-7° (decomposition), and I, m. 263-4° (decomposition).

IT 107225-53-6, Sotetsuflavone, penta-O-methyl-, dioxime (structure of)

RN. 107225-53-6 CAPLUS

CN 3''',8-Biflavone, 4',4''',5,5'',7,7''-hexamethoxy-, dioxime (7CI) (CA INDEX NAME)



L9 ANSWER 47 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1959:17229 CAPLUS Full-text
DOCUMENT NUMBER: 53:17229
ORIGINAL REFERENCE NO.: 53:3203a-e
TITLE: Flavonoids of the leaves of Coniferae and allied plants. I. Flavonoid from the leaves of Torreya nucifera
AUTHOR(S): Kariyone, Tatsuo; Sawada, Tokunosuke
SOURCE: Yakugaku Zasshi (1958), 78, 1010-13
CODEN: YKKZAJ; ISSN: 0031-6903
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

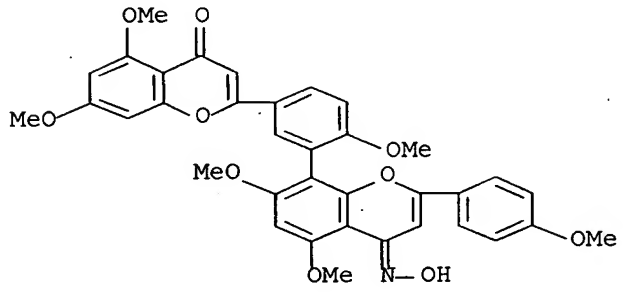
AB Dried leaves (10 kg.) extracted with hot MeOH, the MeOH extract filtered while hot, cooled, the waxy precipitate filtered off, washed with 2% NaOH, the NaOH washing acidified with dilute H2SO4, the precipitate filtered off, washed with C2HCl3, taken up in C5H5N, and H2O added yielded 0.15% kayaflavone (I), C33H24O10.H2O, needles, m. 314-15° (decomposition)(MeOH). I (0.3 g.) and 1.5 g. Ac2O treated with 1-2 drops concentrated H2SO4, the solution poured into H2O, and the precipitate recrystd. from EtOH gave tri-Ac derivative (II) of I, needles, m. 190-1°. I (0.2 g.) in 50 mL. Me2CO, 2 g. MeI, and 2 g. K2CO3 refluxed 1 h., the product concentrated, and recrystd. from Me2CO gave a mono-

Me derivative (III) of I, m. 281-2°, identical with ginkgetin di-Me ether by mixed m.p. Acetylation of III with Ac2O and concentrated H2SO4 gave a diacetate of III, needles, m. 230°, identical with dimethylginkgetin diacetate by mixed m.p. Methylation of I with Me2SO4 gave tri-Me derivative (IV) of I, m. 220-1°, identical with ginkgetin tetra-Me ether by mixed m.p. IV (0.1 g.), 0.1 g. NH2OH.HCl, 50 mg. AcONa, and 3 mL. C5H5N refluxed 5 h., cooled, dilute AcOH added, and the precipitate recrystd. from EtOH gave IV oxime, prisms, m. 250°. Similarly is prepared triethylkayaflavone, columns, m. 236-7°. I (0.5 g.) in a small amount of PhOH and 20 mL. HI heated 2 h. at 130-40°, the solution diluted with H2O, and the precipitate recrystd. from MeOH gave demethylkayaflavone (V), prisms, m. above 330°. Acetylation of V gave V acetate, needles, m. 239-40°. Alkali fusion of I yielded AcOH, p-HOC6H4CO2H, and phloroglucinol. Thus, I is ginkgetin mono-Me ether.

IT 124270-14-0P, Kayaflavone, tri-O-methyl-, oxime
RL: PREP (Preparation)
(preparation of)

RN 124270-14-0 CAPLUS

CN Kayaflavone, tri-O-methyl-, oxime (6CI) (CA INDEX NAME)



L9 ANSWER 48 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:	1958:25516 CAPLUS Full-text
DOCUMENT NUMBER:	52:25516
ORIGINAL REFERENCE NO.:	52:4622f-i,4623a-i,4624a-e
TITLE:	Chemistry of fungi. XXVII. Structure of fulvic acid from Carpenteles brefeldianum
AUTHOR(S):	Dean, F. M.; Eade, R. A.; Moubasher, R.; Robertson, Alexander
CORPORATE SOURCE:	Univ. Liverpool, UK
SOURCE:	Journal of the Chemical Society (1957) 3497-510 CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE:	Journal
LANGUAGE:	Unavailable

GI For diagram(s), see printed CA Issue.

AB cf. C.A. 51, 3507e. Evidence is presented that fulvic acid (I), the yellow acidic metabolite of C. brefeldianum, has the structure indicated. C. brefeldianum was inoculated on Raulin-Thom medium by the method of Oxford, et al. (C.A. 29, 58378), except that a temperature of 26° was used to isolate satisfactory yields of I from the culture medium after an appreciably shorter period (28 days) of growth. The culture fluid acidified, extracted with EtOAc, and the product purified by repeated crystallization from anhyd dioxane (II) yielded I, pale yellow prisms, m. 244° (vigorous decomposition) (softening above 200°), giving a green ferric reaction in alc., λ 225, 318, 343 mμ (log ε 4.47, 4.07, 4.05), with characteristic infrared spectrum. I was recovered from its solution in H2SO4.H2O after being kept overnight and then

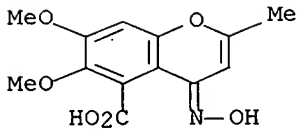
poured on ice. Anhydrofulvic acid (III) (0.2 g.) refluxed 1 hr. with 50 ml. 2N H_2SO_4 and the solution diluted gave 0.13 g. I, m. 244° (II), identified spectroscopically. I (1.5 g.) and 2N aqueous NaOH distilled slowly during 0.5 hr. with the addition of H_2O to maintain the original volume, and the distillate worked up gave 2,4-$(O_2N)_2C_6H_3NHN{:}CMe_2$, yellow needles, m. 124-5°. Acidification of the residual alkaline liquor gave a precipitate which was removed and distillation of the filtrate yielded 0.94 equivalent volatile acids, including HOAc, identified as 2-methylbenzimidazole (IV), m. 173-4°. I (0.25 g.) refluxed 0.5 hr. with 30 ml. 10% aqueous NaOH, the cooled mixture acidified carefully with 2N aqueous H_2SO_4, and warmed gently while a slow stream of N was passed into the mixture and then into $Ba(OH)_2$ solution showed by titration that 0.812 mole of CO_2 had been formed. In similar expts. with III, 0.850 mole CO_2 was formed. Methylation of I with CH_2N_2 or with Me_2SO_4 and Na_2CO_3 (loc. cit.) gave Me di-O-methylfulvate, prisms, m. 186-7° (decomposition) (aqueous II), λ 230, 282, 300 mμ (log ε 4.41, 4.08, 3.99), inflection at 248 mμ (log ε 4.04), insol. in dilute NaOH, and giving a neg. ferric reaction. The solution obtained by heating 1.0 g. Me anhydrodi-O-methylfulvate (VI) 0.75 hr. with 20 ml. MeOH and 20 ml. 2N H_2SO_4, diluted with 40 ml. H_2O, concentrated in vacuo to 35 ml., the precipitate (0.1 g.) filtered off, and the filtrate stored gave 0.85 g. V, m. 191° (decomposition). MeOH (5 ml.) containing NaOMe (from 0.2 g. Na) added to 1.0 g. VI in 10 ml. warm absolute MeOH gave Me tri-O-methylfulvate (VII), cubes, m. 176-7° (C_6H_6-petr. ether), giving no ferric reaction. VI (1.0 g.) and 50 ml. 3% HCl-absolute MeOH gave 0.8 g. VII, m. 175-6° (decomposition), λ 230, 283, 300 mμ (log ε 4.42, 4.09, 4.01). Substitution of EtOH for MeOH in either of the above 2 prepns. gave Me O-ethyldi-O-methylfulvate (VIII), plates, m. 208-9° (decomposition) (EtOH). I (8.0 g.) in 1 1.70% HOAc boiled 10 min. and cooled gave 6.4 g. III, yellow, m. 245-6° (darkening near 235°) (anhydrous or aqueous II), giving a deep green ferric reaction, λ 233, 341, 387 mμ (log ε 4.28, 4.03, 4.34), and characterized by its infrared spectrum. Finely powdered III (10 g.) in 200 ml. Et_2O containing 20 ml. MeOH treated with Et_2O-CH_2N_2 [from 30 g. $Me(NO)NCONH_2$] gave 9.8 g. VI, faint yellow prisms, m. 193-4°, mixed m.p. with V depressed to 170°, mol. weight (micro Rast) 324, λ 235, 257, 318, 348 mμ (log ε 4.12, 4.09, 4.13, 4.20), having a characteristic infrared spectrum. Me_2SO_4 (60 ml.) in 30 ml. MeOH added to a stirred solution of 10 g. III in 200 ml. N aqueous Na_2CO_3, the mixture treated gradually with 400 ml. 2N Na_2CO_3 (the temperature kept below 40°), and the solid collected after 2 hrs., triturated with dilute aqueous NaOH, and crystallized from MeOH gave 9.3 g. VI, m. 193°. Acidification of the alkaline liquors gave a precipitate partly soluble in aqueous $NaHCO_3$, from which solution was obtained 0.1 g. anhydrodi-O-methylfulvic acid, pale yellow plates, m. 219-20° (decomposition) (aqueous MeOH). When sublimed at 180°/0.005 mm., V, VII, and VIII afforded VI, m. 193°. VI (0.5 g.) and 0.5 g. piperonal in 20 ml. warm MeOH containing NaOMe (from 0.2 g. Na) gave, after 2 weeks, 0.4 g. piperonylidene derivative, orange needles, m. 217-18° (decomposition) (MeOH). III (1.0 g.) and $MeCHN_2$ gave 0.5 g. Et anhydrodi-O-ethylfulvate, yellow plates, m. 149-51° (decomposition). VI (3 g.) in 250 ml. EtOAc shaken with 3 g. 10% Pd-C absorbed 240-70 ml. H; the mixture filtered, and the filtrate concentrated to 50 ml. gave 2.3 g. Me deoxydi-O-methylfulvate, prisms, m. 199-200° (EtOAc), giving a neg. ferric reaction, λ 230, 282, 303 mμ (log ε 4.40, 4.08, 4.00). VI (0.5 g.) and 0.5 g. o-$C_6H_4(NH_2)_2$ in 8 ml. EtOH containing 1 ml. HOAc refluxed 1 hr., the solution cooled, 20 ml. H_2O added, the solution treated with C, filtered, and the filtrate neutralized gave 0.6 g. Me 1,2,3,4,9,10-hexahydro-6,7-dimethoxy-2-methyl-10-oxo-9-oxa-3,1'- diazaindeno(2',3'-2,3)-anthracene-5-carboxylate, prisms, m. 209-11° (EtOH) [picrate, plates, m. 263° (decomposition) (HOAc)]. VI (1.0 g.) refluxed 1 hr. with 0.65 g. $HONH_2.HCl$ and 1.4 g. $NaOAc.3H_2O$ in 20 ml. MeOH, the mixture filtered, the filtrate concentrated to 10 ml., diluted with 10 ml. H_2O, the mixture kept several days, and the resulting precipitate (0.15 g.) repeatedly crystallized from EtOH gave Me 6,7-dimethoxy-6'-

methylpyridino(4',3'- 2,3)-chromone-5-carboxylate 1'-oxide, yellow prisms, m. 266° (decomposition), giving a neg. ferric reaction, liberating iodine from HI, λ 242, 282, 326 mμ (log ε 4.08, 4.49, 4.38), characteristic infrared spectrum. N led 0.75 hr. through 3.32 g. VI in 10 ml. boiling II or MeOH containing 80 ml. 2N aqueous NaOH and the effluent gases tested gave neg. tests for carbonyl compds. The solution kept 8 hrs. at 0° deposited 0.5 g. Na salt (IX) and the filtrate from IX gave an addnl. 1.1 g. IX. IX decomposed by acids furnished 1.2 g. 2-acetyl-7-hydroxy-4,5-dimethoxyindan-1,3-dione (X), pale yellow needles, m. 157°, giving a purple-brown ferric reaction, mixed m.p. with so-called 6,7-dimethoxy-2-methylchromone-5-carboxylic acid prepared from citromycetin (XI) 157°; the 2 specimens have identical ultraviolet and infrared spectra, λ 300 mμ (log ε 4.52) [oxime, yellow needles, m. 213-14° (EtOH)]. Methylation of X with MeI and K2CO3 in Me2CO gave the di-Me ether, needles, m. 77°, giving a neg. ferric reaction. After removal of the X, the acidified hydrolyzate was distilled almost to dryness, the distillate treated with KMnO4, the excess KMnO4 destroyed with H2O2 and dilute H2SO4, and the distillate redistd., giving 1.03 ml. HOAc, identified by conversion into IV, m. 175-6°, and into AcNHPh, m. 112°. Ozonolysis of VI gave no definite results. CrO3 in 80% HOAc added dropwise at 50° during 1 hr. to VI, and the mixture kept 1 hr. at 50° and worked up gave Me 6,7-dimethoxy-6'-methyl-α-pyrano[4',3'-2,3]chromone-5-carboxylate, pale cream plates, m. 250° (decomposition), inert toward FeCl3 and [2,4-(O2N)2C6H3NHNH2]2.H2SO4 (XII). KMnO4 (9 g.) added during 2.25 hrs. to 2.5 g. VI in 200 ml. boiling Me2CO, the mixture later clarified by 100 ml. H2O and SO2, concentrated in vacuo to 120 ml., the product extracted with eight 50-ml. portions of Et2O, the extract washed with H2O, extracted with two 25-ml. portions of aqueous NaHCO3 (a separation from 30 mg. neutral nonketonic substance, prisms, m. 206°), the alkaline extract acidified with dilute HCl, the product (XIII) (1.2 g.) isolated with Et2O, the XIII extracted with C6H6, and the insol. material crystallized from aqueous II gave 0.1 g. Me 4-hydroxy-6,7-dimethoxycoumarin-5-carboxylate, cream prisms, m. 255-6° (decomposition), brown ferric reaction; the C6H6 extract allowed to stand and the product which separated recrystd. from C6H6 gave 0.7 g. 6,3,4,2-HO(MeO)2(MeO2C)C6HCO2H, m.p. and mixed m.p. (with sample prepared from XI) 147-8°, characterized by conversion into 6-ethoxy-3,4-dimethoxyphthalic anhydride, m. 195°. Powdered KMnO4 (2.0 g.) added at 0° during 5 hrs. to 2.0 g. VI in 200 ml. Me2CO, the mixture clarified with 100 ml. H2O and SO2, concentrated in vacuo below 40°, extracted with six 25-ml. portions of EtOAc, and the extract washed with H2O and two 25-ml. portions of dilute NaHCO3, and evaporated gave 0.5 g. recovered VI, m. 192° (decomposition); the alkaline extract acidified, and the resulting solid (0.55 g.) leached with 2 10-ml. portions of boiling C6H6 and crystallized from aqueous MeOH gave 3-acetoxymethyl-6,7-dimethoxy-5- carbomethoxychromone-2-carboxylic acid, needles, m. 233-4° (decomposition), giving a neg. test with FeCl3 and XII, λ 221, 314 mμ (log ε 4.39, 3.97), inflections at 235, 292 mμ (log ε 4.35, 3.89) [forming with CH2N2 the Me ester, m. 195° (MeOH)]. VI (0.95 g.) in 10 ml. CHCl3 treated with 1.05 moles BzO2H in CHCl3, the solution extracted after 1 hr. with aqueous NaHCO3, washed with H2O, dried, evaporated, and the residue fractionally crystallized from C6H6 gave Me 2-(1-hydroxyacetonyl)-6,7-dimethoxychromone-5-carboxlate, faint yellow needles, m. 202-4° (decomposition), λ 314 mμ (log ε 3.99); from the fractionation, a very small amount of a compound, m. 237-8° (decomposition) (EtOAc), was also obtained.

IT 100394-34-1
(Derived from data in the 6th Collective Formula Index (1957-1961))

RN 100394-34-1 CAPLUS

CN 4H-1-Benzopyran-5-carboxylic acid, 6,7-dimethoxy-2-methyl-4-oxo-, oxime (6CI) (CA INDEX NAME)



L9 ANSWER 49 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1958:25509 CAPLUS Full-text
DOCUMENT NUMBER: 52:25509
ORIGINAL REFERENCE NO.: 52:4615g-i,4616a-i,4617a-b
TITLE: Vitexin. I
AUTHOR(S): Evans, W. H.; McGookin, A.; Jurd, L.; Robertson, Alexander; Williamson, W. R. N.
CORPORATE SOURCE: Trinity Coll., Dublin, Ire.
SOURCE: Journal of the Chemical Society (1957) 3510-23
CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB cf. Nakaoki, C.A. 46, 108c. Extraction of 20 kg. Vitex littoralis by the method of Perkin (J. Chemical Society 73, 1019(1898)) gave 215 g. vitexin (I), m. 264-5°, [α]20D -14.35 (c 2.414; C5H5N). I (0.2 g.) with 2 ml. Ac2O and 0.5 g. NaOAc at 100° 1 hr. gave vitexin heptaacetate (II), m. 257-8°, [α]20D -73.2° (Me2CO) (neg. ferric test). Similarly, I with 2 g. AcCl in 5 ml. C5H5N gave II. I (10 g.) in 100 ml. C5H5N and 100 ml. Ac2O was heated 2 hrs. on a steam bath, then diluted with 2N HOAc to precipitate vitexin pentaacetate (III), m. 146-7° then 247° (decomposition), [α]20D -4.43°. Addition of excess ethereal CH2N2 to III in methanol gave tetra-O-acetyldi-O-methylvitexin (IV), m. 205-6°, [α]20D -13.52°. IV with NH3 18 hrs. gave di-O-methylvitexin (V), m. 182° then 264°. V with Ac2O-C5H5N on a steam bath 2 hrs. gives IV. Methylation of 4.15 g. III with 40 g. K2CO3 and 40 ml. MeI in 100 ml. boiling Me2CO 8 hrs. gave a gum. The gum in warm C5H5N was treated with hot H2O until precipitation began then cooled to give a product (Va) having a slight ferric reaction. Remethylation of Va gave tetra-O-acetyltri-O-methylvitexin (VI), m. 202°, 212°, [α]20D -9.82°. VI with NH3 gave tri-O-methylvitexin (VII), m. 288°; tetrakis(p-nitrobenzoate), m. 176°. VII in Ac2O-C5H5N gave VI. Similarly, III with K2CO3 and EtI gave tetra-O-acetyltri-O-ethylvitexin (VIII), m. 236°. VIII with NH3 gave tri-O-ethylvitexin (IX), m. 270°. VII (11 g.), 25 g. Ag2O, 40 g. MeI, and 500 ml. Me2CO was refluxed 50 hrs., then evaporated to give a gum which was similarly remethylated 24 hrs. Evaporation gave a gum which was taken up in C6H6. Stepwise addition of petr. ether to the warm solution, then cooling separated an oil (IXa), then a solid, hexa-O-methylvitexin (X), m. 205°, [α]20D -13.45° (MeOH). Addition of the mother liquor to IXa and concentration gave more X, then penta-O-methylvitexin, m. 220° (p-nitrobenzoate, m. 277°). VII was hydrolyzed at reflux with 5% aqueous NaOH under N, then steam distilled giving a product (Xa). Acidification of the residue gave p-anisic acid (XI), m. 182° (amide, m. 164°). A portion of Xa was treated with 2,4-dinitrophenylhydrazine sulfate and the precipitate chromatographed on Al2O3 with C6H6 to give p-methoxyacetophenone 2,4-dinitrophenylhydrazone (XII), m. 256°. Extraction of remaining Xa gave p-MeOC6H4Ac; semicarbazone, m. 198°. VII was hydrolyzed with boiling saturated aqueous Ba(OH)2 under a rapid stream of N. The effluent passed into aqueous 2,4- dinitrophenylhydrazine gave XII. The alkaline residue was acidified and filtered. Extraction of the precipitate with MeOH gave XI. The filtrate was treated with BaCO3, filtered, and evaporated to give di-O-methylapovitexin (XIII), C14H16O7(OMe2), m. 126-30°, then 222° (decompose), [α]20D -4.59°; pentakis-(p-nitrobenzoate), m. 192°; pentaacetate, m. 151-2°. Aqueous HIO4

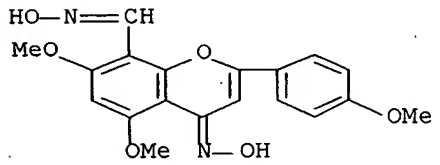
(10 ml. 6%) was added to 0.2 g. XIII in 10 ml. HOAc, the mixt agitated 5 hrs., diluted with 100 ml. H2O, then kept 16 hrs. to give 3-formyl-2-hydroxy-4,6-dimethoxyacetophenone (XIV), m. 170°; 2,4-dinitrophenylhydrazone, m. 259°. Reduction of 4 g. XIV with 10 g. Zn-Hg in 30 ml. HOAc and 8 ml. concentrated HCl 2-3 min. and dilution with H2O gave 2.5 g. 2-hydroxy-4,6-dimethoxy-3-methylacetophenone, m. 144°. Hydrolysis of IX with Ba(OH)2 gave di-O-ethylapovitexin, m. 203.5-204°; pentakis(p-nitrobenzoate), m. 175°. VII (1.5 g.), 4.5 g. Pb(OAc)4, and 25 ml. HOAc was kept at 25° 5 days, poured into H2O, extracted with CHCl3, the extract washed with NaHCO3, dilute NaOH, and H2O, then evaporated to give 8-formyl-5,7,4'- trimethoxyflavone (XV), m. 237°; 2,4-dinitrophenylhydrazone, m. 250-4°; dioxime, m. 232°. Hydrogenation of XV over Raney Ni in dioxane gave 5,7,4'-trimethoxy-8-methylflavone (XVI), m. 230°. 2-Hydroxy-4,6-dimethoxy-3-methylacetophenone (13 g.), 12 g. p-MeOC6H4COCl, and 75 ml. C5H5N was heated 3 hrs. on a steam bath, cooled, and poured into 500 ml. H2O to give 16 g. 2-p-anisoyloxy-4,6-dimethoxy-3- methylacetophenone (XVII), m. 169°. XVII (7 g.) was refluxed with 35 g. NaNH2 in 75 ml. C6H6 4 hrs. to give 4 g. 2-hydroxy-4,4',6-trimethoxy- 3-methyldibenzoylmethane (XVIII), m. 184°; monoxime, m. 188°. Cyclization of XVIII with 75% H2SO4 10 min. at room temperature gave XVI. VII (0.3 g.), 5 ml. HNO3, and 25 ml. H2O was refluxed 1.5 hrs., cooled, filtered, basified, and extracted with CHCl3 to give XV. VII (1 g.) in 150 ml. MeOH was treated with 35 ml. 0.5N HIO4, the mixture kept 10 hrs. in the dark, filtered, neutralized with Ba(OH)2, filtered, the filtrate evaporated, and the residue extracted with CHCl3 to give 0.1 g. XV. A stirred solution of 2 g. VII in 40 ml. HOAc was treated with 70 ml. 0.5N HIO4 added in 10 ml. portions at 1 hr. intervals, the mixture diluted with excess saturated aqueous NaHCO3, kept 4 days at room temperature, and filtered. Extraction of the precipitate with boiling MeOH gave 0.9 g. dehydro-O-methylsecovitexin (XIX), m. 197-8°; bis(p-nitrobenzoate), m. 159-60°. XIX (0.2 g.) in 10 ml. MeOH, 10 ml. H2O, and 2 ml. H2SO4 gave a distillate containing MeCOCHO, isolated as the 2,4-dinitrophenylosazone, m. 299-300°, and the phenylosazone, m. 145°. I (8.3 g.) in 1 l. MeOH and 300 ml. H2O was treated with 15 g. NaIO4 in 1 l. H2O, kept 20 hrs., and filtered to give 4.2 g. dehydrosecovitexin (XX), m. above 360°, [α]20D -147° (C5H5N); pentaacetate, m. 242°, [α]20D -68.25° (HOAc); pentakis(p-nitrobenzoate), m. 225°. Concentration of the filtrate from XX and extraction with Et2O gave 8-formylapigenin (XXI), m. 301°. Methylation of XXI gave XVI. Distillation of XX in the same manner as XIX also gave MeCOCHO. XX (10.1 g.), 350 ml. MeOH, and 10 ml. H2SO4 was refluxed 2.5 hrs., diluted with 1.5 l. Et2O and washed several times with aqueous NaHCO3. The Et2O was evaporated and the residue dissolved in Me2CO. The solution was concentrated until precipitation began, then cooled to precipitate 1.1 g. C18H12O6(OMe)2, m. above 360°, [α]20D 90.5°; tris(p-nitrobenzoate), m. 277°; di-Me ether (XXII), m. 247-9°, [α]20D -86.3° (MeOH). XXII gave a p-nitrobenzoate, m. 145°. The Me2CO-filtrate was diluted with EtOAc and the Me2CO evaporated On standing, 3.5 g. C18H12O6(OMe)2.H2O precipitated, m. 188-90°, [α]20D -118°; tris(p-nitrobenzoate), m. 254-5°; di-Me ether (XXIII), m. 251-2°, [α]20D -68.5°. XXIII gave a p-nitrobenzoate, m. 124-6°. XX (2.0 g.), 80 ml. MeOH, and 2 ml. H2SO4 was refluxed 2 hrs. then poured into excess BaCO3. After 0.5 hr. the filtered mixture was treated with more BaCO3, filtered, and evaporated in vacuo. The residue was extracted with warm Me2CO. The extract was evaporated and the residual oil in C5H5N treated with p-O2NC6H4COCl to give D-glyceraldehyde di-Me acetal bis(p-nitrobenzoate), m. 104-6°, [α]18D -59.6° (CHCl3). XIII (0.5 g.) in 100 ml. H2O was treated with 0.32 g. NaIO4 in 50 ml. H2O, the solution kept overnight, and the H2O removed by successive concns. and addns. of Me2CO then C6H6. After 5 days was isolated 0.34 g. dehydrodi-O- methylsecoapovitexin (XXIV), m. 158-9°, [α]23D -30.7°. Hydrolysis of 0.72 g. XXIV in 32.3 ml. MeOH and 0.82 ml. H2SO4 at reflux 0.75 hr. gave 3-acetyl-2-hydroxy-4,6-dimethoxyphenylacetaldehyde, m. 114-16°.

IT 102174-83-4

(Derived from data in the 6th Collective Formula Index (1957-1961))

RN 102174-83-4 CAPLUS

CN 4H-1-Benzopyran-8-carboxaldehyde, 5,7-dimethoxy-2-(p-methoxyphenyl)-4-oxo-, dioxime (6CI) (CA INDEX NAME)



L9 ANSWER 50 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1958:6368 CAPLUS Full-text

DOCUMENT NUMBER: 52:6368

ORIGINAL REFERENCE NO.: 52:1151i,1152a-i,1153a-c

TITLE: Chromane. V. A new synthesis of khellin and other furo-2-methylchromones

AUTHOR(S): Dann, Otto; Illing, Gerhard

CORPORATE SOURCE: Univ. Erlangen, Germany

SOURCE: Ann. (1957), 605, 146-57

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 52:6368

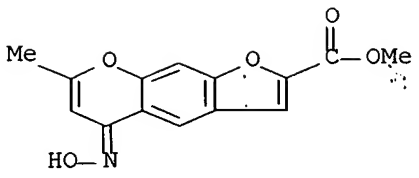
AB cf. C.A. 50, 2564c, 2566c, 2570f; 51, 6943d. The difficulties in preparing khellin (I) (2-methyl-5,8-dimethoxyfuro-2',3'; 7,6-chromone) are reviewed. 2,4-Dihydroxy-3,6-dimethoxybenzaldehyde (0.1 mole) in 250 cc. dry Me2CO was stirred with 0.36 mole K2CO3 with the dropwise addition of 0.1 mole PhCH2Br, refluxed 3 hrs., 0.36 mole K2CO3 added, and 0.11 mole BrCH2CO2Me added dropwise, the mixture refluxed 8 hrs., filtered hot, washed with Me2CO, evaporated, refluxed 4 hrs. with 10 g. Mg filings and 350 cc. dry MeOH, treated with ice, 2N HCl added at 0°, and stirred with 1 l. H2O. The product, m. 102°, was debenzylated by AcOH or MeOH giving 4,7-dimethoxy-6-hydroxy-2-carbomethoxycoumarone (II) (cf. Baxter, et al., C.A. 44, 155h). II refluxed in C6H6 with AcCl and Mg gave 91% 6-Ac derivative of II, m. 122° which treated with anhydrous HF gave II. 6-Hydroxy-2-carbomethoxycoumarone (IIa) (0.01 mole) was stirred 2 hrs. at 100° with 0.01 mole cis-MeCCl:CHCO2H in 30 cc. H3PO4 and 50 g. P2O5, poured into ice H2O, filtered from 0.5 g. resin, and the filtrate extracted with CHCl3 giving 0.87 g. mixture (III), m. poorly 213-18°, giving in poor yield with HONH2.HCl and pyridine 2-methyl-5'-carbomethoxy- 2',3'; 5,6(or 2',3';7,6)-chromonoxime, C14H11O5N, m. 205° (aqueous MeOH). III crystallized repeatedly from EtOH and then from MeOH gave 4-methyl-5'-carbomethoxyfuro-2',3',5,6(or 2',3';7,6)-coumarin (IV), C14H10O5, λmaximum 255 and 290 mμ (log ε 4.6 and 4.19), λmin. 283 mμ (log ε 4.14), giving no oxime. IIa (0.01 mole) and 0.01 mole cis-MeCCl:CHCO2Me refluxed and stirred 8 hrs. in 100 cc. dry Me2CO containing 0.075 mole K2CO3 gave Me β-(5'-carbomethoxyfuro-2',3';3,4-phenoxy)crotonate (V), m. 138° (EtOH). V (2.9 g.) in 30 cc. H3PO4 containing 60 g. P2O5 kept 1 hr. at 20° and stirred 5 hrs. at 70°, decomposed dropwise with ice H2O, and the filtrate extracted with CHCl3 gave 0.78 g. IV. 6-Hydroxycoumaran (VI) (1.36 g.) and 0.86 g. MeCH:CHCO2H in 30 cc. HF kept 2 days at 20° and shaken occasionally gave 0.8 g. 6-hydroxy-5-crotonylcoumaran, yellow, m. 121° (aqueous MeOH), which in little MeOH with 1% NaOH gave 2-methyl-4',5'-dihydrofuro- 2',3';7,6-chromanone, m. 94° (aqueous MeOH); oxime, m. 206° (EtOH). VI and trans-MeCCl:CHCO2H in HF gave 35% 6-hydroxy-5-(β-chlorocrotonyl)coumaran, yellow, m. 114°, which in 1.5% NaOH yielded 2-methyl-

4'5'-dihydrofuro-2',3';7,6-chromone (VII), m. 164° (H2O). As in the preparation of V, VI and trans-MeCCl:CHCO2Me gave 85% Me β-(4',5'-dihydrofuro-2',3';3,4-phenoxy)crotonate, m. 81° (aqueous MeOH); free acid (VIII), m. 180° (with loss of CO2). VIII with AcCl and a few drops H2SO4 stirred, freed from excess AcCl, and poured into ice H2O gave VI, m. 166-7°. Formed by methods analogous to those described were: Me β-(2,5-dimethoxy-4',5'-dihydrofuro-2',3';3,4- phenoxy)crotonate (IX), m. 134° (prepared from 4,7-dimethoxy-6-hydroxycoumaran); free acid, C14H16O6 (X), m. 190° (decomposition). 4-Methyl-4',5'-dihydrofuro-2',3';7,6-coumarin m. 167° (m. 143° when mixed with VII), λmaximum 225 and 230 mμ (log ε 4.08 and 4.18), λmin. 265 mμ (log ε 3.16). IX (2 g.) in 70 cc. absolute Et2O at -5° stirred with 2 g. MeCN and 4 g. dry ZnCl2, cooled below 0°, saturated with dry HCl, and kept 5 days gave a precipitate which was washed with Et2O and decomposed with 250 cc. H2O at 90° giving 1.8 g. 5-Ac derivative of IX (dihydrokhellinone), pale yellow, m. 105°. X (0.9 g.) with AcCl and H2SO4 gave 0.25 g. 4',5'-dihydro derivative of I, m. 151°. H2SO4 (85%) (10 cc.) at 0° added dropwise to 1 g. IX and 0.7 cc. AcCH2CO2Et and kept 48 hrs. at 20° gave a precipitate which was purified by solution in 2N NaOH and precipitation with HCl giving 1.3 g. 4-methyl-5-hydroxy-6-methoxy-4',5'- dihydrofuro-2', 3';7,8(or 7,6)-coumarin, C13H12O5, m. 270°, λmaximum 328 mμ (log ε 4.09), λmin. 278 mμ (log ε 3.12) (EtOH), λmaximum 320 mμ (log ε 4.21), λmin. 2.57 mμ (log ε,3.21) (MeOH), which with EtI and K2CO3 in dry MeOHMe2CO gave the Et ether, C15H16O5, m. 114° (H2O). Formed analogously to V was 75% Me β-(furo-2',3';3,4- phenoxy)crotonate, m. 45-6° (75% MeOH); free acid, m. 179° (decomposition), cyclized gave 2-methylfuro-2',3';5,6-chromone, m. 225° (MeOH); oxime, m. 139° (decomposition). 1,3,2,5-(HO)2(MeO)2C6H2 (68 g.) in 1 l. absolute Et2O stirred below 0° with 55 g. anhydrous ZnCl2 and 30 g. ClCH2CN, then saturated with dry HCl, stirred 24 hrs., and the resulting precipitate washed with Et2O and warmed with 0.5 l. H2O gave 2,4-dihydroxy-3,6- dimethoxy-ω-chloroacetophenone, m. 148°, which was kept 5 hrs. in 750 cc. N NaOH, cooled to 0°, and acidified with concentrated HCl giving 65 g. 4,7-dimethoxy-6-hydroxy-3-coumaranone (XI), m. 178-80° (after washing with ice H2O, and drying in vacuo), turning red in air. A similar reaction carried out with 1,4,2,6-(MeO)2(PhCH2O)2C6H2 gave the 6-PhCH2 derivative of XI, m. 123°; oxime, m. 194-5° (decomposition). The oxime (XII) of XI darkened on heating and decomposed about 198°. XII (30 g.) slurried at 40-50° with 600 cc. EtOH and 80 cc. glacial AcOH was treated gradually with 1.5 kg. 2.5% Na-Hg and concomitantly, dropwise, with enough glacial AcOH to insure an acid mixture, stirred 12 hrs. at 20°, decanted, and the residue washed with H2O. All solns. were mixed and evaporated in vacuo; the residue was refluxed 2 hrs. with 500 cc. H2O, and this solution extracted with Et2O; the dried, evaporated extract gave 14 g. 4,7-dimethoxy-6-hydroxycoumarone, b1.2 145°, n22D 1.5721, rapidly turning orange, 5.83 g. of which with 5 g. trans-MeCCl:CHCO2Me in Me2CO with K2CO3 gave a product which on attempted distillation at 3 mm. decomposed at 210°, and which, saponified (without distillation) gave 58% β-(2,5-dimethoxyfuro-2',3';3,4-phenoxy)crotonic acid, m. 189° (decomposition), 1.8 g. of which with 15 cc. AcCl and 5 drops H2SO4 after 10 days gave 0.52 g. I, m. 152-3°. 20 references.

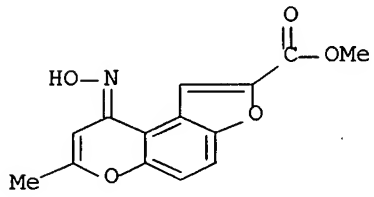
IT 107558-98-5P, 5H-Furo[3,2-g][1]benzopyran-2-carboxylic acid, 7-methyl-5-oxo-, methyl ester, oxime 110060-05-4P, 9H-Furo[2,3-f][1]benzopyran-2-carboxylic acid, 7-methyl-9-oxo-, methyl ester, oxime 116055-72-2P, 9H-Furo[2,3-f][1]benzopyran-9-one, 7-methyl-, oxime
RL: PREP (Preparation)
(preparation of)

RN 107558-98-5 CAPLUS

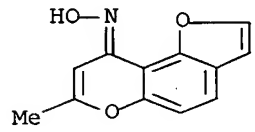
CN 5H-Furo[3,2-g][1]benzopyran-2-carboxylic acid, 7-methyl-5-oxo-, methyl ester, oxime (6CI) (CA INDEX NAME)



RN 110060-05-4 CAPLUS
CN 9H-Furo[2,3-f][1]benzopyran-2-carboxylic acid, 7-methyl-9-oxo-, methyl ester, oxime (6CI) (CA INDEX NAME)



RN 116055-72-2 CAPLUS
CN 9H-Furo[2,3-f][1]benzopyran-9-one, 7-methyl-, oxime (6CI) (CA INDEX NAME)



L9 ANSWER 51 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1957:21784 CAPLUS Full-text
DOCUMENT NUMBER: 51:21784
ORIGINAL REFERENCE NO.: 51:4401b-h
TITLE: The constituents of Casimiroa edulis. I. The seed
AUTHOR(S): Kincl, F. A.; Romo, J.; Rosenkranz, G.; Sondheimer, Franz
CORPORATE SOURCE: Syntex S. A., Mexico D. F., Mex.
SOURCE: Journal of the Chemical Society (1956) 4163-9
CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

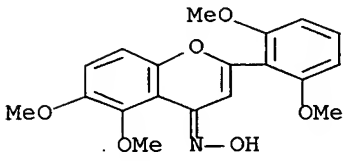
AB cf. Power and Callan, C.A. 6, 667. The dried, ground kernels were extracted twice with 400 l. hot EtOH. The combined exts. were evaporated and diluted with 50 1.4% aqueous HCl. The mixture was extracted (5 + 10 l. each) with C6H14 (A), C6H6 (B), CH2Cl2 (C), and AmOH (D). The aqueous layer was basified with aqueous NH3 and extracted similarly to give the basic exts. (E, F, G, and H), resp. A was chromatographed on 40 parts of Al2O3. Elution with 7:3 C6H6-Et2O gave β-sitosterol, m. 138-9°, [α]D -38°; acetate, m. 127-8°, [α]D -38°; benzoate, m. 145-7°, [α]D -12°. Further elution with 1:1 C6H6-Et6O gave

palmitamide, m. 103-4°. Chromatography of B and elution with 4:1 C6H6-Et6O gave zapotin, C19H18O6 (I), m. 150-1 (picrate, m. 181-2°; perchlorate, m. 204-6°; oxime, m. 240-3°); 3:1 C6H6-Et2O gave casimiroin, C12H11NO4 (II), m. 202-3° (picrate, m. 193-4°; aurichloride, m. 196-8°); 4:1 Et2O-EtOAc gave N-benzoyltyramine (III), m. 161-2° (acetate, m. 121-2°; benzoate, m. 172-3°). I (4 g.) was refluxed 1 hr. with 60 ml. Ac2O and 85 ml. aqueous HI to yield 3.1 g. of demethylzapotin, C15H10O6 (IV), m. 321-5°, green with alc. FeCl3. KOH-fusion of IV yielded salicylic acid, m. 156-8°, and resorcinol (dibenzoate, m. 116.5°). Refluxing II 20 min. in 20% aqueous HCl gave casimiroinol, C11H9NO4 (V), m. 321-3°. V with CH2N2 gave II. KOH-fusion of III gave BzOH. III with CH2N2 gave the Me ether, m. 123-4° which was oxidized with alkaline KMnO4 to give p-MeOC6H4CO2H. C yielded 9-hydroxy-4- methoxyfurano[3,2-g]benzopyran-7-one (VI), m. 223-4°; acetate, m. 181-2° benzoate, m. 203-5°. VI with CH2N2 gave isopimpinellin, C13H10O5, m. and mixed m.p. 150-1°. VI with CrO3-AcOH gave bergaptenquinone, m. 251-3° (decomposition). VI in alkaline KMnO4 gave 2,3-furandicarboxylic acid, m. 220-1°. Chromatography of the mother liquor from C and elution with C6H6 gave zapotinin, C18H16O6 (VII), m. 224-5° green with alc. FeCl3 (acetate, m. 214-16°); C6H6-CH2Cl2 gave zapoterin, C19H24O6 (VIII), m. 257-9° [α]D -51°; CH2Cl2 gave casmirolid, m. 229-31, [α]D -49°. KOH-fusion of I at 270° for 20 min. gave VII. IV with CH2N2 also gave VII. VIII kept 1 hr. with Ac2O and C5H5N at 90° gave isozapoterin, m. 284-5°. D separated β-sitosterol β-D-glucoside, m. 290-5° (decomposition); tetraacetate, m. 164-6°. Chromatography of F and elution with 9:1 C6H6-Et2O gave eduline, C17H15-NO2, m. 187-8°; picrate, m. 225-7° perchlorate, m. 250-2°. Chromatography of G and elution with C6H6 gave zapotidine, C7H9N3S, m. 96-8°; picrate, m. 195-6°. H crystallized casimiroedine, C17H24N2O5, m. 224-5°, [α]D -33°.

IT 111441-11-3
(Derived from data in the 6th Collective Formula Index (1957-1961))

RN 111441-11-3 CAPLUS

CN Zapotin, oxime (6CI) (CA INDEX NAME)



L9 ANSWER 52 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1957:21783 CAPLUS Full-text

DOCUMENT NUMBER: 51:21783

ORIGINAL REFERENCE NO.: 51:4401a-b

TITLE: Alkaloid studies. XIV. The structure of the cactus alkaloid pilocereine

AUTHOR(S): Djerassi, Carl; Figdor, S. K.; Bobbitt, J. M.; Markley, F. X.

CORPORATE SOURCE: Wayne Univ., Detroit, MI

SOURCE: Journal of the American Chemical Society (1956), 78, 3861-2
CODEN: JACSAT; ISSN: 0002-7863

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

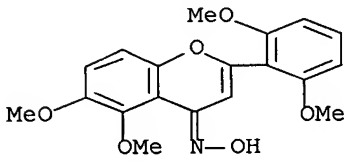
GI For diagram(s), see printed CA Issue.

AB In an abstract of this paper (C.A. 51, 1217f), the lower half of formula I should be as follows:

IT 111441-11-3
(Derived from data in the 6th Collective Formula Index (1957-1961))

RN 111441-11-3 CAPLUS

CN Zapotin, oxime (6CI) (CA INDEX NAME)



L9 ANSWER 53 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1956:89182 CAPLUS Full-text

DOCUMENT NUMBER: 50:89182

ORIGINAL REFERENCE NO.: 50:16759g-i,16760a

TITLE: Structure of sciadopitysin, a flavonoid from the leaves of Sciadopitys verticillata. IV. Degradation of sciadopitysin trimethyl ether in ethanolic potassium hydroxide solution

AUTHOR(S): Kawano, Nobusuke

SOURCE: Yakugaku Zasshi (1956), 76, 457-61
CODEN: YKKZAJ; ISSN: 0031-6903

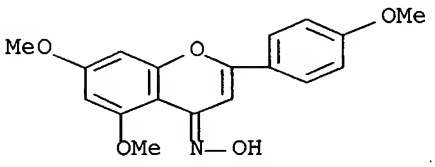
DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

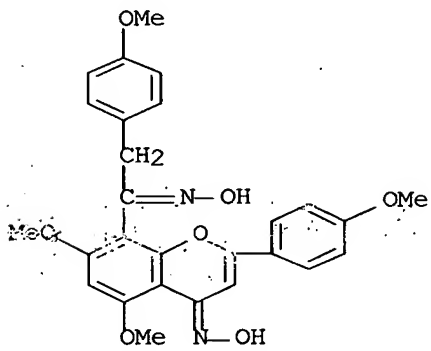
AB IIIa (1 g.) and 15 mL. 15% KOH-EtOH refluxed 3.5 h., the solution acidified with H2SO4, the precipitate filtered off and boiled 30 min. with 20 mL. 30% KOH, 80 mL. water added, the mixture extracted with Et2O, and the extract evaporated gave p-MeOC6H4Ac (semicarbazone, m. 198-9°); the mother liquor extracted with Et2O and saturated with CO2 and the precipitate filtered off and recrystd. from EtOH gave 50 mg. putative 5,2,4,6-Ac(MeO)2(HO)C6HCOCH2C6H4OH-x (VII), plates, C19H20O6, m. 226-7°; the mother liquor from VII extracted with Et2O gave 10 mg. 6,2,4-HO(MeO)2C6H2Ac, m. 79-80°; this mother liquor evaporated, the residue acidified with H2SO4, the precipitate (VIIa) filtered off, and the filtrate extracted with Et2O gave 10 g. anisic acid, m. 179-81°. VIIa and 5 mL. Me2CO concentrated, 2 mL. MeOH added, and the mixture allowed to stand gave 10 mg. putative 6,2,4-HO(MeO)2C6H2COCH2C6H3(OMe)CO2H-x,x' (VIII), columns, m. 293-4°. The mother liquor from VIII evaporated to dryness, the residue extracted with CCl4 and recrystd. from dilute EtOH gave 6,2,4-HO(MeO)2C6H2CO2H, m. 156-8° (decomposition). IV di-Me ether (400 mg.) and 6 mL. 15% KOH-EtOH refluxed 40 min. and the product treated as in the preparation of VII gave 20 mg. p-MeOC6H4Ac, 15 mg. VII, and 15 mg. putative 6,5,2,4-HO(HO2C)(MeO)2C6HCOCH2C6H4OMe-x (IX), m. 217-18° (decomposition), and 8 mg. anisic acid. VII forms a monoxime, C19H21O6N, m. 230-2°; Ac derivative of VII, C21H22O7, m. 169° (dioxime, C21H24O7N2, m. 207°). Me ester of VIII, C19H20O7, m. 202°; mono-Me ether (VIIIa) of VIII, C19H20O7, m. 203° (decomposition); VIIIa oxime, C19H21O7N, m. 231°. Ac derivative of VIII, C20H20O8.H2O, m. 119-20° (oxime, C20H21O8N, m. 242°). It is suggested that I is 5-hydroxy-2-[5-hydroxy-7-methoxy-2-(4-methoxyphenyl)-4-oxo-4H-1-benzopyran-8-yl]-3-(4-hydroxyphenyl)-7-methoxychromone.

IT 872302-08-4P, Flavone, 4',5,7-trimethoxy-, oxime
RL: PREP (Preparation)
(preparation of)

RN 872302-08-4 CAPLUS
CN Flavone, 4',5,7-trimethoxy-, oxime (5CI) (CA INDEX NAME)



L9 ANSWER 54 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1956:89181 CAPLUS Full-text
DOCUMENT NUMBER: 50:89181
ORIGINAL REFERENCE NO.: 50:16759f-g
TITLE: Structure of sciadopitysin, a flavonoid from the leaves of Sciadopitys verticillata. III. The structure of oxoflavone and carboxyflavone
AUTHOR(S): Kariyone, Tatsuo; Kawano, Nobusuke
SOURCE: Yakugaku Zasshi (1956), 76, 453-6
CODEN: YKKZAJ; ISSN: 0031-6903
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB IV and V were each shown to have 2 MeO groups and 2 phenolic HO groups and were assumed to have the skeleton of acacetin 7-Me ether (VI) with an acyl group as the side-chain. IV is VI where the acyl is COCH2C6H4OH and V is IV bearing a group on the VI moiety.
IT 874530-27-5P, Flavone, 4',5,7-trimethoxy-8-[(p-methoxyphenyl)acetyl]-, dioxime
RL: PREP (Preparation)
(preparation of)
RN 874530-27-5 CAPLUS
CN Flavone, 4',5,7-trimethoxy-8-[(p-methoxyphenyl)acetyl]-, dioxime (5CI) (CA INDEX NAME)



L9 ANSWER 55 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1956:89180 CAPLUS Full-text
DOCUMENT NUMBER: 50:89180
ORIGINAL REFERENCE NO.: 50:16759e-f

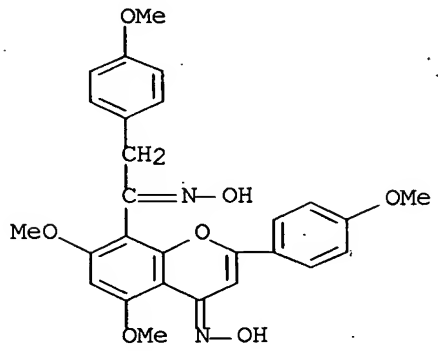
TITLE: Structure of sciadopitysin, a flavonoid from the leaves of Sciadopitys verticillata. II. Degradation of sciadopitysin
AUTHOR(S): Kariyone, Tatsuo; Kawano, Nobusuke
SOURCE: Yakugaku Zasshi (1956), 76, 451-2
CODEN: YKKZAJ; ISSN: 0031-6903
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB Decomposition of I by boiling 1.5 hrs. with 20% aqueous KOH afforded an oxoflavone (IV), C25H20O7, yellow columns, m. 241-2°, a carboxyflavone (V), C26H20O9, yellow, m. 311° (decomposition), anisic acid, p-MeOC6H4Ac, and 4,2,6-MeO-(HO)2C6H2Ac; the yield of these products varied with the concentration of KOH and duration of boiling.

IT 874530-27-5P, Flavone, 4',5,7-trimethoxy-8-[(p-methoxyphenyl)acetyl]-, dioxime
RL: PREP (Preparation)
(preparation of)

RN 874530-27-5 CAPLUS

CN Flavone, 4',5,7-trimethoxy-8-[(p-methoxyphenyl)acetyl]-, dioxime (5CI) (CA INDEX NAME)



L9 ANSWER 56 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1956:89179 CAPLUS Full-text
DOCUMENT NUMBER: 50:89179
ORIGINAL REFERENCE NO.: 50:16759d-f
TITLE: Structure of sciadopitysin, a flavonoid from the leaves of Sciadopitys verticillata. I
AUTHOR(S): Kariyone, Tatsuo; Kawano, Nobusuke
SOURCE: Yakugaku Zasshi (1956), 76, 448-50
CODEN: YKKZAJ; ISSN: 0031-6903
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

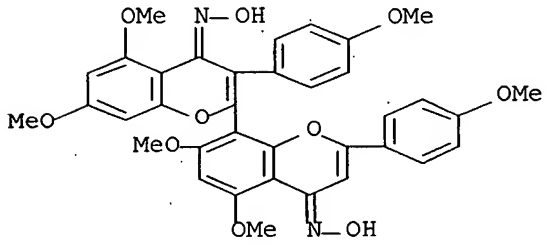
AB Air-dried leaves (3 kg.) in 20 l. CHCl:CCl2 refluxed 3 hrs., the extract concentrated to 3 l., allowed to stand overnight, the waxy precipitate warmed up, and the insol. residue filtered off gave 9 g. sciadopitysin (I), yellow columns, m. 285-6° (from C5H5NEtOH); triacetate, prisms, m. 264°; tri-Me ether (IIIa), m. 214-15° (oxime, C36H32O10N2, columns, m. 248-9°). I (0.1 g.) in 40 ml. Me2CO treated with 0.5 g. each of K2CO3 and MeI and the mixture refluxed 40 min. and concentrated gave a I mono-Me ether (II), m. 282°, identical with ginkgetin di-Me ether (III) by mixed m.p. II gives a diacetate, m. 228°, identical with the diacetate of III by mixed m.p. Demethylation of I with HI gave C30H18O10.5H2O, m. above 360°, and acetylation of this substance gave a

product, m. 240°, identical with that prepared by treating ginkgetin in a similar way.

IT 854209-81-7P, [2,8'-Bi-4H-1-benzopyran]-4,4'-dione, 5,5',7,7'-tetramethoxy-2',3-bis(p-methoxyphenyl)-, dioxime
874530-27-5P, Flavone, 4',5,7-trimethoxy-8-[(p-methoxyphenyl)acetyl]-, dioxime
RL: PREP (Preparation)
(preparation of)

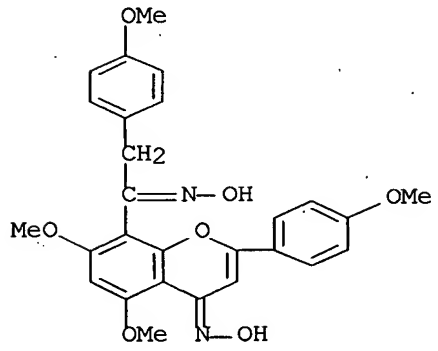
RN 854209-81-7 CAPLUS

CN [2,8'-Bi-4H-1-benzopyran]-4,4'-dione, 5,5',7,7'-tetramethoxy-2',3-bis(p-methoxyphenyl)-, dioxime (5CI) (CA INDEX NAME)



RN 874530-27-5 CAPLUS

CN Flavone, 4',5,7-trimethoxy-8-[(p-methoxyphenyl)acetyl]-, dioxime (5CI) (CA INDEX NAME)



L9 ANSWER 57 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1956:12315 CAPLUS Full-text

DOCUMENT NUMBER: 50:12315

ORIGINAL REFERENCE NO.: 50:2570f-i,2571a-i,2572a-i,2573a-f

TITLE: Synthesis of chromanones, chromans, and 2-methylchromones with hydrofluoric acid

AUTHOR(S): Dann, Otto; Volz, Gerda; Huber, Otto

CORPORATE SOURCE: Univ. Erlangen, Germany

SOURCE: Ann. (1954), 587, 16-37

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB A number of substances related structurally to certain portions of the rotenone (I) mol. have been synthesized and tested as insect and as fish

poisons. Action of HF on mixts. of phenols and acrylic acids (or on Ph acrylates) gives chromanones, which are reduced by the Clemmensen method to the corresponding chromans; use of β-chloropropionic acid (II) gives substances unsubstituted in the 2-position. Chromones are similarly prepared employing cis-β-chlorocrotonic acid (III). Ph esters (except those of II) are prepared by the procedure of Spasov (C.A. 36, 7010.2). p-Cresol (IV) crotonate (100 g., prepared in 90% yield, b15 133° nD23 1.523) is heated in a cast steel bomb with 100 mL. com. anhydrous HF for 3 h. at 100°; pouring the reaction product into ice H2O gives a brown oil which soon solidifies and is then taken up in min. hot EtOH, poured into 1.5 l. 1.5% aqueous NaOH, and shaken overnight to give 60% 2,6-dimethylchromanone (V), m. 52-4° (from petr. ether), which turns deep yellow on treatment with concentrated H2SO4. Similar treatment of a mixture of IV and crotonic acid (VI) (heated with HF 2 h. at 100°) gives 82% V, b11 135-7°. V (8 g.) in 80 mL. HOAc mixed with 200 g. amalgamated Zn dust and 300 mL. 18% HCl and let stand 24 h. at room temperature, 80 mL. 12% HCl added, the mixture refluxed 6-7 h., cooled, and extracted with Et2O, and the extract distilled gives 2,6-dimethylchroman (VII), b11 115-25° nD16 1.531. Resorcinol (VIII) dicrotonate (in HF at 18° for 3 h.) gives 60% 3-crotonoyloxy-4-crotonoylphenol (IX), deep yellow needles from C6H6, m. 138° FeCl3 test (in MeOH) deep violet; on standing overnight, the deep orange solution of IX in 1.5% NaOH lightens in color and on acidification yields 72% 7-hydroxy-2-methylchromanone (X), m. 175-6° (from C6H6). VIII and VI (with HF, 2 h. at 100°, followed by treatment with 1.5% NaOH) give X in 84% yield. The action of Me2SO4 and alkali on X (2.5 g.) gives 0.6 g. 7-methoxy-2-methylchromanone (XI), m. 74-7°. Reduction of + with amalgamated Zn and HCl gives 68% 7-hydroxy-2-methylchroman (XII), b0.5 120-5°, m. 67-8°, pale yellow with concentrated H2SO4. XII is converted by Me2SO4 and alkali to 7-methoxy-2-methylchroman (XIII) (1.5 g. from 2 g.), b12 130-50°, nD13 1.539. Heating p-cresol β-chloropropionate (36 g., prepared in 40% yield, b12 145-50°) in HF for 3 h. at 55-60° and distillation of the crude product (b11 145-55°) gives 16 g. alkali-soluble material which is refluxed 20 h. in 20% aqueous Na2CO3 to give 2,5-dimethylcoumaranone, b11 140-50°, m. 52-4° (from petr. ether), FeCl3 test neg.; oxime, m. 129°; semicarbazone, m. 191°. IV and II (20 g. each) heated 5 h. in HF at 50° gives 11 g. 2-(β-chloropropionyl)-p-cresol, yellow, m. 60-2° (from MeOH), which gives 52% 6-methylchromanone (XIV), b0.5 100-20°, m. 36° (from petr. ether), on treatment with 1.5% aqueous NaOH. Heating II and VIII in HF for 1.5 h. at 50° gives 33% 4-(β- chloropropionyl)resorcinol yellow, m. 96° (from petr. ether-C6H6), converted with 1.5% NaOH to 81% 7-hydroxychromanone, m. 146° (from EtOAc). Action of alkali on a mixture of o-cresol and II gives a 26% yield of crude β-(2-methylphenoxy)propionic acid, m. 92-3°, heating of which with HF for 5 h. at 55-6° gives 64% 8-methylchromanone (XV) (distilled in vacuo, nD22 1.572); reduction of 3 g. XV with Zn-HCl gives 0.4 g. 8-methylchroman (XVI), nD20 1.541. β-(2,5- Dimethylphenoxy)propionic acid is similarly prepared (yield 16%), needles from 50% aqueous HOAc, m. 108-10°, and converted to 5,8-dimethylchromanone (XVIA, 60%), pale yellow, b1.5 110-30° nD20 1.567; oxime, m. 116-17° (from MeOH); with 3 g. XVIA with Zn-HCl gives 0.8 g. 5,8-dimethylchroman (XVII), b5 90-110°, nD20 1.543. Similarly, 3.5 g. β-(2,3-dimethylphenoxy)propionic acid (prepared in 25% yield) gives 1.6 g. reddish 7,8-dimethylchromanone (XVIII), b3 120-40°, m. 47-8.5° [oxime, m. 165-6° (from petr. ether-C6H6)]; 1.6 g. XVIII gives 0.4 g. 7,8-dimethylchroman (XIX), b2 120-40°, nD23 1.541. HF converts β-(2,3,5- trimethylphenoxy)propionic acid (m. 118-20°, prepared in 10% yield) to 5,7,8-trimethylchromanone (XX, yield 76%), b2 100-20° (congeals to waxy solid), orange in concentrated H2SO4 [oxime, m. 142-3° (from petr. ether-C6H6)], reduced to 59% 5,7,8-trimethylchroman (XXI), b4 100-20°, m. 53° colorless in concentrated H2SO4. Heating 5 g. m-cresol crotonate (b12 135-7°, nD14 1.522, m. about 20°, prepared in 70% yield) in HF for 3 h. at 100° gives 3 g. 2,7-dimethylchromanone (XXII), b12 150-60°, nD16 1.557, citron-yellow in

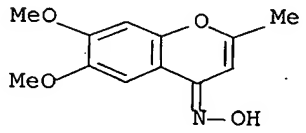
concentrated H_2SO_4 [oxime, m. 140° (from MeOH)], reduced (0.4 g. from 1.5 g.) to 2,7-dimethylchroman (XXIII), b0.5 100-10°. 3,4-Dimethylphenol (12.2 g.) and 8.6 g. VI in HF (2 h. at 100°) give 12 g. 2,6,7-trimethylchromanone (XXIV), m. 90-1° [oxime, m. 176-7° (from petr. ether-C_6H_6)]; reduction of 5 g. XXIV gives 2 g. 2,6,7-trimethylchroman (XXV), nD^{20} 1.526, which solidifies on standing. Heated 3 h. at 100° in HF, 10 g. 3,5-dimethylphenol crotonate (b12 138-40°, prepared in 85% yield) gives 4 g. yellow 2,5,7-trimethylchromanone (XXVI), m. 65°, citron-yellow in concentrated H_2SO_4 [oxime, m. 140° (from MeOH)]; on reduction, 2.5 g. XXVI yields 1.5 g. 2,5,7-trimethylchroman (XXVII), nD^{22} 1.528. 2,6,8- Trimethylchromanone (XXVIII), brownish needles from aqueous MeOH, m. 59-60° [oxime, m. 124-5° (from petr. ether-C_6H_6)], is obtained in 63% yield by heating 2,4-dimethylphenol with VI in HF for 2 h. at 100°; XXVIII is reduced to 43% 2,6,8-trimethylchroman (XXIX), m. 44-5°. Similarly, 6.8 g. 2,3,5-trimethylphenol (XXX) and 4.3 g. VI condense to give 5.8 g. 2,5,7,8-tetramethylchromanone (XXXI), b4 140-60°, m. 47-8° [oxime, m. 151-2° (from petr. ether-C_6H_6)]. Reduction (with ZnHCl) of 2.5 g. XXXI gives 0.9 g. 2,5,7,8-tetramethylchroman (XXXII), m. 46-8° (from aqueous MeOH). Hydroquinone dicrotonate (recrystd. from MeOH, m. 112-14°, difficultly soluble in C_6H_6) is the precursor of yellow 6-hydroxy-2- methylchromanone, m. 152-3° (from C_6H_6), orange in concentrated H_2SO_4, converted by treatment with Me_2SO_4 and alkali (but not with CH_2N_2) to 6-methoxy-2-methylchromanone (XXXIII), m. 65-7° (from C_6H_6), insol. in alkali, $FeCl_3$ test neg.; Zn-HCl reduction of 3 g. XXXIII gives 1.5 g. 6-methoxy-2-methylchroman (XXXIV), nD^{25} 1.529. Heating 5 g. 2,4-dihydroxytoluene (XXXV) with 3.5 g. VI in HF for 2 h. at 100° gives 6 g. crude brown 7-hydroxy-2,6-dimethylchromanone (XXXVI), m. 194-6° after precipitation from EtOAc by addition of petr. ether; oxime, m. 172-4° (from EtOAc diluted with petr. ether). Action of Me_2SO_4 and alkali on XXXVI gives 7-methoxy-2,6-dimethylchromanone (XXXVII), m. 116-17°. Reduction of 6 g. XXXVI with Zn-HCl gives 2.5 g. 7-hydroxy-2,6-dimethylchroman (XXXVIII), m. 126-7° (from HOAc diluted with H_2O), which is converted to 7-methoxy-2,6-dimethylchroman (XXXIX), m. 53-4° (from HOAc diluted with H_2O), by treatment with Me_2SO_4-alkali. Similarly, 5 g. 2,6-dihydroxytoluene (XL) yields 7.5 g. crude 7-hydroxy-2,8-dimethylchromanone, m. 175-6° after precipitation from EtOAc with petr. ether; oxime, m. 194-6°, purified in the same way), which is methylated (with MeI and K_2CO_3 in acetone) to 7-methoxy-2,8-dimethylchromanone (XLI), m. 80-1° (petr. ether); and 6 g. reduced (with Zn-HCl) to 2.3 g. crude 7-hydroxy-2,8-dimethylchroman (XLII), needles from petr. ether, m. 71-3°, which is converted by the action of Me_2SO_4 and alkali to 7-methoxy-2,8-dimethylchroman (XL-III), b0.5 95-100°, nD^{18} 1.539. Treating 5 g. 3,4-dimethoxyphenol crotonate (b0.8 149-50°, m. 35-40°) with HF at room temperature for 6 h., followed by treatment with 1.5% aqueous NaOH, gives 3.2 g. crude 6,7-dimethoxy-2-methylchromanone (XLIV), m. 117-18° (from petr. ether), deep red in concentrated H_2SO_4 [oxime, m. 172-4° (from MeOH on addition of H_2O)]; reduction of 2.7 g. XLIV gives 1.3 distilled 6,7-dimethoxy-2- methylchroman (XLV), nD^{20} 1.545, which solidifies, m. 47-8°. Similarly, 5 g. trimethylhydroquinone dicrotonate (yellow, m. 124° from petr. ether) yields (after 3 h. at 100° in HF) 1 g. 6-hydroxy-2,5,7,8-tetramethylchromanone (XLVI), yellow needles from C_6H_6, m. 111-12° orange in concentrated H_2SO_4; oxime, needles from petr. ether-C_6H_6, m. 151-3°. XLVI (5 g. crude) is also obtained by heating 7.8 g. trimethylhydroquinone and 4.3 g. VI in HF for 2.5 h. at 100°. Methylation (Me_2SO_4-alkali) of 0.6 g. XLVI yields 0.2 g. 6-methoxy-2,5,7,8-tetramethylchromanone (XLVII), needles from MeOH, m. 81-2°, Zn-HCl reduction of which gives 6-methoxy-2,5,7,8- tetramethylchroman (XLVIII), b0.2 140-60°, which congeals to a yellow solid, m. 53-4°. Although reaction with CH_2N_2 or with MeI and K_2CO_3 in acetone was unavailing, prolonged treatment of dl-α-tocopherol with Me_2SO_4 and alkali gave the corresponding Me ether (XLIX), yellow oil, b0.005 200°, $nD^{20.5}$ 1.4995. p-Chlorophenol crotonate (b13 150-2°, nD^{18} 1.535, prepared in 83% yield) (10 g.) in HF at 100° for 3 h. gives 2 g. 6-chloro-2-methylchromanone (L), m. 98-9° (from petr. ether); oxime, needles, m. 147° (from MeOH). IV and 1- cyclohexenecarboxylic acid

(LI) in HF for 2 h. at 100° give 6-methyl-2,3-cyclohexanochromanone (6-Methyl-2,3-tetramethylenechromanone) (LII), m. 82.5-5° (from aqueous MeOH); oxime, m. 168-70° (from MeOH). Reduction of 0.9 g. LII gives 0.3 g. 6-methyl-2,3-cyclohexanochroman (LIII), which solidifies after distillation Similarly, 6 g. XXXVI and 6.1 g. LI give 10 g. crude 7-hydroxy-6-methyl-2,3-cyclohexanochromanone, converted by treatment with Me2SO4 and 20% aqueous KOH to 2.2 g. yellow 7-methoxy-6-methyl-2,3-cyclohexanochromanone (LIV), m. 144-5° (from MeOH); oxime, m. 189-90° (from petr. ether-C6H6). XXX (6.8 g.) similarly yields 10 g. crude 5,7,8-trimethyl-2,3-cyclohexanochromanone (LV), distillation of which (b4 140-70°) gives a solid m. body temperature [oxime, m. 198-200° (from petr. ether-C6H6)]; reduction of 6 g. crude LV gives 1.2 g. 5,7,8-trimethyl-2,3-cyclohexanochroman (LVI), m. 66-8°. In the same way, 8.1 g. VIII and 10 g. LI yield 15 g. crude yellow 7-hydroxy-2,3-cyclohexanochromanone, treatment of which with Me2SO4-20% KOH gives 7-methoxy-2,3-cyclohexanochromanone (LVII), needles from MeOH, m. 114-15°; oxime, m. 174-5° (from petr. ether-C6H6). LVII is reduced to 7-methoxy-2,3-cyclohexanochroman (LVIII), a wax melting near body temperature XII is converted to the corresponding crotonate, b0.4 150-80°, which solidifies and is recrystd. from petr. ether, needles, m. 76-8° (yield 66% from XII); treatment of 4 g. of this with HF at 100° for 3 h. followed by chromatog. on Al2O3 and distillation of the crude product, gives a viscous red-yellow oil, b0.5 180-90°, which solidifies and is recrystd. from petr. ether tc give 0.8 g. 2-methylchromanono[6,7:α,β]-γ-pyran-α'- methyl-α',β'-dihydride, C14H16O3, (LIX), m. 82-5°; oxime, m. 151-3° (from petr. ether-C6H6). Distillation of the crude product obtained by treatment of 10 g. β-naphthol crotonate (b12 197-8°, m. 46.5-8°, prepared in 76% yield) with HF at room temperature for 3 h. gives a solid recrystd. from petr. ether to yield 2 g. violet-tinged 2-methyl-5,6-benzochromanone (LX), m. 73-4°; oxime, m. 200-4° (from petr. ether-C6H6). LX is reduced to 2-methyl-5,6-benzochroman (LXI), b4 120-40°, m. 88-90°. p-Cresol cis-β-chlorocrotonate (b1 136-8° nD20 1.532) is treated with HF at 100° for 3 h. and the crude product run through a column of Al2O3 to give 70% 2,6-dimethylchromone, yellow needles from petr. ether, m. 98-100°, which condenses with piperonal in the presence of NaOMe to give yellow 6-methyl-2-(piperonylidenemethyl)chromone , m. 193-5° from EtOAc. VIII and III (m. 61°) react in HF at 18° (8 h.) to give 65% 4-(cis-β-chlorocrotonyl)resorcinol, yellow needles from petr. ether-C6H6, m. 114-16°, Beilstein and FeCl3 tests pos.; cyclization is effected with 1.5% aqueous NaOH to give 62% 7-hydroxy-2-methylchromone, m. 250° from EtOH. Similarly, 5 g. each of XXXVI and III (after 1.5 days in HF at room temperature and treatment of the crude product with 1.5% NaOH overnight) gives 4 g. 7-hydroxy-2,6-dimethylchromone, pale rose crystals from MeOH, m. 259-60°, which is treated with MeI and K2SO3 in acetone to give pale yellow 7-methoxy-2,6-dimethylchromone, recrystd. from EtOAc and sublimed at 150-70°/0.4 mm., m. 129-30°; oxime, m. 225-6° (from MeOH). Similarly, XL and III (20 h. in HF at room temperature) give 72% 7-hydroxy-2,8-dimethylchromone (LXII), pale rose needles from MeOH, m. 255-60°, difficultly soluble in Et2O or acetone; oxime, yellow, m. 178-9° (from MeOH on addition of H2O). Treatment of 1.5 g. LXII with MeI and K2CO3 gives 1.0 g. 7-methoxy-2,8-dimethylchromone, yellow needles from EtOAc, m. 141-2°. Similarly, distillation (b0.6 190-200°) of the crude product from 3,4-dimethoxyphenol and III, followed by recrystn. from MeOH, gives 0.5 g. 6,7-dimethoxy-2- methylchromone, m. 146-7°; oxime, m. 205-6° (from MeOH). Reaction of 3.8 g. XII with 3 g. III in HF at room temperature for 24 h. and treatment of the crude product with 1.5% NaOH gives 1.2 g. 2,6'-dimethyl-γ-pyrono[2',3':6,7]chroman, needles from MeOH, m. 130°, the "linear" structure of which is established by comparison of its UV spectrum (in MeOH) with those of 2,6',8-trimethyl-γ- pyrono[2',3':6,7]chroman (LXIII) and 2,6,6'-trimethyl-γ-pyrono[2',3':7,8]chroman (LXIV). LXIII(needles, m. 196-7° 0.9 g.) is prepared from 5 g. XLII and 3.5 g. III; while 2 g. XXXVIII and 1.3 g. III yield 0.2 g. yellow LXIV, m. 115-16° from petr. ether. In a fly (Musca domestica) killing

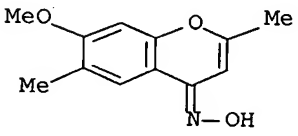
test similar to that of Wagner-Jauregg (C.A. 43, 2945a), XI, XXIV, XXVI, XXVIII, XXXI, XXXIII, XXXVII, XLI, XLIV, XLV, XLVII, XLIX, L, LII, LIV, LV, LVI, LVII, LVIII, LIX, LX, LXI, and 7-methoxy-6-methyl-2,3-cyclohexanochroman are inactive; active (like I, at 0.1 g.) are chroman itself, 6-chloro-2-methylchroman, and dihydrodeoxyrotenone; active at 0.01 g. are unsubstituted chromanone, 6-methylchroman, V, VII, XIV, XV, XVI, XVIA, XVII, XVIII, XIX, XX, XXII, XXIII, XXV, XXVII, XXIX, XXXIV, XLIII, XLVIII, and LIII; active at 0.001 g. are XIII, XXI, XXXII, and XXXIX. These activities are to be compared with that of γ-hexachlorocyclohexane (LXV), which is active in the same test at 10-5 g. In the test of Spath and Kuffner (C.A. 31,761.3) (against Lebistus reticulatus), the fish toxicity of V, XI, and XIII at 0.1 g. is equivalent to that of LXV at 10-3 g. or to that of I at 10-4 g.

IT 854845-38-8P, Chromone, 6,7-dimethoxy-2-methyl-, oxime
854846-08-5P, Chromone, 7-methoxy-2,6-dimethyl-, oxime
854846-33-6P, Chromone, 7-hydroxy-2,8-dimethyl-, oxime
RL: PREP (Preparation)
(preparation of)

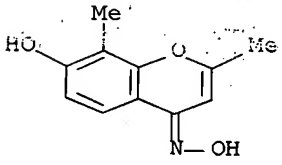
RN 854845-38-8 CAPLUS
CN Chromone, 6,7-dimethoxy-2-methyl-, oxime (5CI) (CA INDEX NAME)



RN 854846-08-5 CAPLUS
CN Chromone, 7-methoxy-2,6-dimethyl-, oxime (5CI) (CA INDEX NAME)



RN 854846-33-6 CAPLUS
CN Chromone, 7-hydroxy-2,8-dimethyl-, oxime (5CI) (CA INDEX NAME)



L9 ANSWER 58 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1956:4711 CAPLUS Full-text
DOCUMENT NUMBER: 50:4711
ORIGINAL REFERENCE NO.: 50:978a-i

TITLE: Synthesis of nuclear-substituted flavonoids and allied compounds. V. Structure of the flavone formed by degradation from ginkgetin. 2. Syntheses of 8-(2-anisoyl-ethyl)4',5,7-trihydroxyflavone methyl ethers

AUTHOR(S): Nakazawa, Koichi; Matsuura, Shin

SOURCE: Yakugaku Zasshi (1955), 75, 68-71
CODEN: YKKZAJ; ISSN: 0031-6903
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

GI For diagram(s), see printed CA Issue.

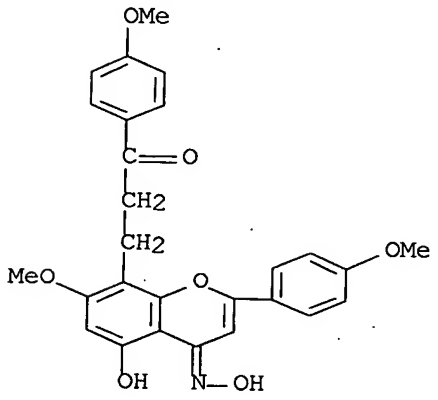
AB cf. C.A. 49, 1714e. EtOH (1 mL.), 5 mL. dioxane, and 46 mg. Na treated with 0.44 g. p-MeOC6H4COCH2CO2Et and 0.53 g. 8-chloromethyl-7- methoxyacacetin, heated 30 min. on a water bath, acidified with AcOH, diluted with water, the upper solution decanted, the precipitate in the lower layer allowed to stand 6 h. with 5 mL. each of dioxane and 10% KOH, the product diluted with water, and the precipitate filtered and recrystd. from AcOEt gave 0.4 g. 8-(2-anisoyl-ethyl)-5-hydroxy-4',7-dimethoxyflavone (I), yellow needles, m. 198°; the filtrate acidified with AcOH, and the precipitate filtered and recrystd. from AcOEt gave 0.17 g. I; 100 mg., I and 50 mg. each of NH2OH.HCl and AcONa in 2 mL. C5-H5N heated 3 h. at 110° gave 50 mg. I oxime, needles, m. 225°; 0.22 g. I and 4 mL. Me2SO4 treated with 40% KOH portionwise yielded 0.12 g. 8-(2-anisoylethyl)-4',5,7- trimethoxyflavone (II), granules, m. 152°. 2,4,6-HO(MeO)2C6H2CH2CH2CO2H (IIIa) (3.4 g.), 20 mL. MeCN, and 80 mL. dry Et2O treated with 20 g. ZnCl2, dry HCl gas passed in, the resulting solid kept 10 days in a sealed container, the Et2O removed, the residue taken up in water, the solution made weakly acid with NH4OH, washed with Et2O, boiled 30 min. and the product recrystd. from dilute AcOH gave 2.1 g. 2,4,6,3-HO(MeO)2(HO2CCH2CH2)C6HAc (III), needles, m. 179°; methylation of 0.3 g. III with 0.2 g. each of Me2SO4 and K2CO3 in 20 mL. Me2CO by refluxing 1.5 h., evaporating the solution to dryness, and recrystg. the residue from ligroine gave 0.2 g. Me ester (IV) of III, prisms, m. 147°; 0.56 g. IV in 10 mL. C5H5N heated 2 h. at 110° with 2 g. p-MeOC6H4COCl, cooled, allowed to stand 30 min. with EtOH, the solvent removed in vacuo, 20% HCl added, and the product filtered and washed with water and 10% K2CO3, gave 0.5 g. p-methoxybenzoate (V) of IV, needles, m. 121°; 0.42 g. V, 0.18 g. NaNH2, and 10 mL. xylene heated 30 min. at 110°, the product filtered hot, washed with C6H6 and Et2O, taken up in water, and CO2 passed in yielded 0.15 g. 2,3,4,6-HO(p-MeOC6H4COCH2CO)(MeO)2C6HCH2CH2CONH2(VI), yellow; the filtrate acidified with AcOH gave 30 mg. 2,3,4,6-HO(p-MeO C6H4 CO CH2CO)(MeO)2C6HCH2CH2CO2H (VII). VI (50 mg.) in 3 mL. AcOH and 1 mL. concentrated H2SO4 heated 5 min. at 100° and the product diluted with water gave 45 mg. 8-(2-carbamoylethyl)-4',5,7-trimethoxyflavone (VIII), needles, m. 282°. Cyclization of 30 mg. VII as above yielded 25 mg. 8-HO2CCH2CH2 analog (IX) of VIII, columns, m. 259°; or saponification of 100 mg. VIII in 3 g. AcOHH2SO4-H2O (2:2:1) 1.5 h. at 110° and dilution with water gave 70 mg. IX, m. 259°. IX (100 mg.) 2 drops PhOMe, and 2 g. (HPO3)n (n = 2.5) heated 30 min. at 100°, water added, the volatile substances driven off by passing in steam, and the residue recrystd. from CCl4 gave 6 mg. 8-(p-MeOC6H4COCH2CH2) analog of VIII, granules, m. 143°. Methylating 0.5 g. IIIa in 2 mL. each of Me2SO4 and MeOH in a strongly alkaline solution in 40% NaOH, acidifying the solution with HCl, and recrystg. the product from ligroine gave 0.4 g. 2,4,6-(MeO)3C6H2CH2CH2CO2H (X), needles, m. 140°; methylating 0.5 g. III as above, acidifying, and recrystg. the product from ligroine gave 0.45 g. 3,2,4,6-HO2CCH2CH2(MeO)3C6HAc (XI), needles, m. 116°. X (1.2 g.), 0.5 g. PhOMe, and 15 g. (HPO3)n heated 30 min. at 100°, the product decomposed with water, steam passed in, and the residue recrystd. from ligroine gave 0.45 g. 2,1,3,5-(p-MeOC6H4COCH2CH2) C6H2(OMe)3 (XII), leaves, m. 118 °; or 0.6 g. XI, 0.3 g. PhOMe, and 10 g. (HPO3)n treated as above gave XII. IIIa (0.4 g.), 0.2 g. AcOH, and 5 g. (HPO3)n heated 20 min. at 100° for acetylation, the product decomposed with water, and the

precipitate washed with water and recrystd. from MeOH gave 0.3 g. of the lactone, 3,5-(MeO)2C6H2.CH2.CH2.CO.O, needles, m. 105°.

IT 859441-00-2P, Flavone, 8-(2-p-anisoylethyl)-5-hydroxy-4',7-dimethoxy-, oxime
RL: PREP (Preparation)
(preparation of)

RN 859441-00-2 CAPLUS

CN Flavone, 8-(2-p-anisoylethyl)-5-hydroxy-4',7-dimethoxy-, oxime (5CI) (CA INDEX NAME)



L9 ANSWER 59 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1954:46252 CAPLUS Full-text
DOCUMENT NUMBER: 48:46252
ORIGINAL REFERENCE NO.: 48:8226a-d
TITLE: Synthesis of biologically active new chromone derivatives
AUTHOR(S): Vargha, L.; Rados, M.
CORPORATE SOURCE: Pharm. Research Inst., Budapest
SOURCE: Acta Chimica Academiae Scientiarum Hungaricae (1953), 3, 223-9
CODEN: ACASA2; ISSN: 0001-5407
DOCUMENT TYPE: Journal
LANGUAGE: German

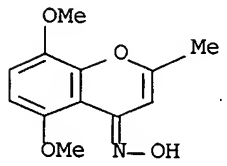
AB 2,3,6-HO(MeO)2C6H2COCHAc (I) obtained in 65% yield by treating 4.6 g. Na powder with 11 g. 2,3,6-HO(MeO)2C6H2Ac (II), 150 ml. absolute EtOH, and 6.4 g. absolute MeOH, m. 112-14° (from alc.). I (4.8 g.) in 50 ml. absolute EtOH treated with 2 ml. concentrated HCl and the product purified in vacuo gives 4 g. of a labile oxonium salt (III), m. 158-60°; III (4 g.) heated 15 min. in 150 ml. dioxane gives 3.5 g. 2-methyl-5,8-dimethoxychromone (IV), m. 129-30°; oxime, m. 107-8.5°. 2,3,4- HO(MeO)2C6H2CH2COCO2Et (V), obtained in 26 g. yield by treating 19.6 g. II and 43.8 g. (CO2Et)2 with 6.9 g. Na in 300 ml. absolute EtOH and triturating the Na salt with 10% HOAc, m. 85-7° (from H2O). 5,8-Dimethoxychromone-2-carboxylic acid (VI) Et-ester (VII), obtained in 90% yield by heating 29.6 g. V in 150 ml. glacial HOAc with 8 ml. concentrated HCl, m. 173-4° (from alc.). VI, obtained in 70% yield by heating 27.8 g. VII 6 hrs. in 150 ml. glacial HOAc with 200 ml. 4N H2SO4, m. 230-1° (from HOAc), forms no oxime. Bu ester of VI obtained in 60% yield from 2.5 g. VI, 250 ml. BuOH, and 20 g. concentrated H2SO4 refluxed 6 hrs., diluted with HOH, and neutralized with NaHCO3, m. 95-6° (from 60% MeOH), forms no oxime. 6,7-Dimethoxychromone-2-carboxylic acid (C.A. 44, 7317a) (25 g.), in 400 ml. BuOH

and 140 g. concentrated H2SO4 refluxed 8 hrs., diluted with HOH, and neutralized with NaHCO3, gives 18.5 g. Bu ester, m. 131-2.5° (from 75% alc.). The presence of the MeO groups enhances the pharmacol. activity of the chromone derivs. similar to IV, but has little effect if a carboxyl group is already present; the position of the MeO groups seems to be unimportant.

IT 854846-47-2P, Chromone, 5,8-dimethoxy-2-methyl-, oxime
RL: PREP (Preparation)
(preparation of)

RN 854846-47-2 CAPLUS

CN Chromone, 5,8-dimethoxy-2-methyl-, oxime (5CI) (CA INDEX NAME)



L9 ANSWER 60 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1950:49331 CAPLUS Full-text

DOCUMENT NUMBER: 44:49331

ORIGINAL REFERENCE NO.: 44:9441h-i,9442a-d

TITLE: The structure of ginkgetin, a flavone derivative from the leaves of ginkgo trees

AUTHOR(S): Nakazawa, Koichi

CORPORATE SOURCE: Gifu Coll. Pharmacy

SOURCE: Yakugaku Zasshi (1941), 61, 228-9
From: Complete Abstracts Japan. Chem. Lit. 15, 883-5(1941).
CODEN: YKKZAJ; ISSN: 0031-6903

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

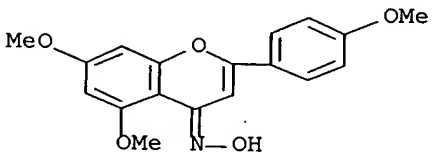
AB An alc. extract of the fallen leaves taken up with ether, extracted with 10% K2CO3, the precipitated K compound decomposed with acid, and the product recrystd. from Me2CO gives ginkgetin (I). The Me2CO-insol. portion also gives free I; yield, 0.02-0.03%. Recrystn. of I from Me2O gives yellow-white needles, m. 297°, giving a brown-purple color with alc.-FeCl3, orange-red with Mg-concentrated HCl, yellow with concentrated H2SO4, insol. in alkali bicarbonates, soluble in alkali carbonates, but precipitating on cooling. I has 2 MeO, 4 OH, and 2 CO groups (does not form an oxime). I has the composition C32H22O10 and mol. weight 572; tetraacetate, C32H18O6(OAc)4, m. 258°, gives no color with FeCl3; di-Me ether, C32H20O8(OMe)2, m. 282°, gives a brown-purple color with FeCl3, insol. in alkali (diacetate, m. 228°); tetra-Me ether, C32H18O6(OMe)4, m. 228°, gives no color with FeCl3 (oxime, m. 250°); tetra-Et ether, m. 175°, mol. weight 700°. Demethylation of I gives C30H18O10, m. 330°, whose acetate, C30H12O4(OAc)6, m. 239-40°. In general the m. ps. of I or its derivs. are 50-60° higher than those of apigenin-type compds. (acacetin, genkwanin, etc.). Fusion of I with KOH give p-HOC6H4CO2H, AcOH, phloroglucinol, and an acid, C9H10O5 (2,4,6-MeO(HO)2C6H2CH2CO2H), m. 188° (decomposition). I with alkaline KMnO4 gives no definite oxidation product but the di-Me ether gives anisic acid. Heating I with 20% aqueous KOH 3.5 hrs. gives water-soluble p-HOC6H4Ac and a water-insol. compound, C24H18O7 (II), m. 280°, easily soluble in organic solvents, gives a dark-green color with alc.-FeCl3 [semicarbazone, m. 268° (decomposition)]. The above expts. indicate that I is composed of 2 mols. of apigenin 7-Me ether (III) (genkwanin) with the suggested union binding at 3,8' or 3,6'. For comparison

acacetin di-Me ether, C16H10O3(OMe)2, m. 156-7°, was prepared from acacetin, Me2SO4, and KOH; oxime, C18H17O5N, m. 140°; di-Et ether, m. 194°. Methylation of II with MeI and K2CO3 gives a di-Me ether, C23H13O4(OMe)3, m. 224°.

IT 872302-08-4P, Flavone, 4',5,7-trimethoxy-, oxime
RL: PREP (Preparation)
(preparation of)

RN 872302-08-4 CAPLUS

CN Flavone, 4',5,7-trimethoxy-, oxime (5CI) (CA INDEX NAME)



L9 ANSWER 61 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1938:41824 CAPLUS Full-text

DOCUMENT NUMBER: 32:41824

ORIGINAL REFERENCE NO.: 32:5833g-i,5834a

TITLE: The synthesis of 5-hydroxy-6-aminoflavone

AUTHOR(S): Sugasawa, S.

SOURCE: Yakugaku Zasshi (1936), 56, 105-7
From: Chem. Zentr. 1936, II, 3669
CODEN: YKKZAJ; ISSN: 0031-6903

DOCUMENT TYPE: Journal

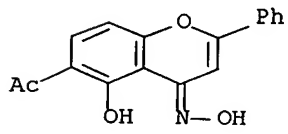
LANGUAGE: Unavailable

AB cf. C. A. 28, 6717.7; 29, 787.8. In some earlier expts. on the synthesis of primetin (cf. C. A. 28, 1345.5; 29, 160.9) work was done with 5-hydroxy-6-acetylflavone (cf. Baker, C. A. 29, 1422.4). Its oxime was caused to undergo the Beckman rearrangement to give 5-hydroxy-6- aminoflavone (I). Attempts to replace the NH2 by OH are as yet unsuccessful. 5-Hydroxy-6-acetylflavone oxime, C17H13O4N, gives yellow needles from glacial HOAc, m. 237-8°. When 1 g. of this compound is introduced into 10 cc. well cooled POCl3, warmed 3-5 min. at 70-80° and poured onto ice, yellowish brown hair-like crystals of 5-hydroxy-6-acetaminoflavone, C17H13O4N, m. 234-5° (from 1:1 alc.-glacial HOAc) are obtained. I is obtained by boiling this product 1.5-2 h. with about 20% HCl and decomposing the precipitate (probably the HCl salt) with aqueous Na2CO3; crystals from alc., m. 177°. I is golden yellow, its sulfate and HCl salt are white and difficultly soluble in water.

IT 854244-53-4P, Flavone, 6-acetyl-5-hydroxy-, oxime
RL: PREP (Preparation)
(preparation of)

RN 854244-53-4 CAPLUS

CN Flavone, 6-acetyl-5-hydroxy-, oxime (4CI) (CA INDEX NAME)

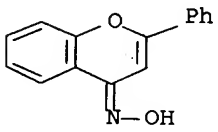


L9 ANSWER 62 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1936:61866 CAPLUS Full-text
DOCUMENT NUMBER: 30:61866
ORIGINAL REFERENCE NO.: 30:8214c-d
TITLE: A new method of oximation
AUTHOR(S): Gulati, K. C.; Ray, J. N.
SOURCE: Current Science (1936), 5, 75
CODEN: CUSCAM; ISSN: 0011-3891
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB cf. C. A. 29, 163.5. The oximes of flavone and α-naphthaflavone were obtained by reaction with NH2OH in aqueous pyridine as follows: reflux 0.1 g. 4 hrs. with 0.15 g. NH2OH.HCl in 0.5 cc. H2O and 1 cc. pyridine, and pour into dilute AcOH when cold. Crystallized from hot dilute acetone, flavone gave colorless needles, m. 237°, and α-naphthaflavone colorless needles, m. 181°.

IT 22115-89-5P, Flavone, oxime
RL: PREP (Preparation)
(preparation of)

RN 22115-89-5 CAPLUS
CN 4H-1-Benzopyran-4-one, 2-phenyl-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 63 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1936:22405 CAPLUS Full-text
DOCUMENT NUMBER: 30:22405
ORIGINAL REFERENCE NO.: 30:2946g-i,2947a-b
TITLE: Hydroxycarbonyl compounds. X. Coumarins and chromones from m-4-xylenol
AUTHOR(S): Flynn, Daniel G.; Robertson, Alexander
SOURCE: Journal of the Chemical Society (1936) 215-17
CODEN: JCSOA9; ISSN: 0368-1769
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

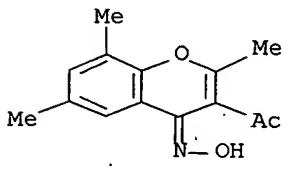
AB cf. C. A. 28, 4419.1. -m-4-Xylenol (I) and AcCH2CO2Et with 86% H2SO4 yield 4,6,8-trimethylcoumarin, m. 114-14.5°; this yields β-3,5-trimethylcinnamic acid, m. 139°, oxidation of which yields 3,5,2-Me2(MeO)C6H2Ac, whose semicarbazone m. 193°. 3,4,6,8-Tetramethylcoumarin yields 2-methoxy-α,β,3,5-tetramethylcinnamic acid, m. 139.5-40°. 4,6,8-Trimethyl-3- ethylcoumarin, m. 112.5-13°; 2-methoxy-β,3,5-trimethyl-α- ethylcinnamic acid, m. 112°. I and BzCH2CO2Et with 86% H2SO4 give 4-phenyl-6,8-di-methylcoumarin, m. 111°; PhCH2CH2CO2Et gives 3-benzyl-4,6,8-trimethylcoumarin, m. 112-13°. 3,5,2-Me2(HO) C6H2Ac, AcOEt and Na, heated 5.5 hrs. on the water bath, give 2-hydroxy-β-acetyl-3,5-dimethylacetophenone, m. 85°; AcOH-HCl yields 2,6,8-trimethylchromone, m. 125°; this was also prepared from I, AcCH2CO2Et and P2O5 on heating 3 hrs.; condensation with piperonal gives 2-(3',4'-methylenedioxystyryl)-6,8-dimethylchromone, pale yellow, m. 195°. The propionate of I, b20 124-5°, and AlCl3, heated at 130-40° for 5 hrs., give 2-hydroxy-3,5-dimethylpropiophenone, m. 52-3° (deep blue FeCl3 reaction); Ac2O and AcONa give 2,3,6,8-tetramethylchromone, m. 136-7°; this also results from

I and AcCHMeCO2Et with P2O5: the 2-(3',4'-methylenedioxystyryl) derivative, yellow, m. 196°. The butyrate of I b17.5 132-3°; 2-hydroxy-3,5-dimelhylbutyrophenone, b30 145-50°, m. 30° (84% yield) (deep blue FeCl3 reaction); acetylation gives 2,6,8-trimethyl-3-ethylchromone, m. 112.5°, which also results from I and the requisite ester; the 2-(3',4'-methylenedioxystyryl) derivative, pale yellow, m. 202-3°. 3,5,2-Me2(HO)C6H2Ac (II), Ac2O and AcONa give 2,6,8-trimethylchromone, m. 125°, and the 3-Ac derivative (oxime, m. 119°). Refluxing II and EtCO2Et with Na for 3 hrs. gives 6,8-dimethyl-2-ethylchromone, m. 109-10°. Heating II with (EtCO)2O and EtCO2Na at 210° for 10.5 hrs. gives 3,4',6,8-tetramethylcoumarin and 3-propionyl-6,8-dimethyl-2-ethylchromone (oxime, m. 93°). Heating II with Bz2O and BzONa gives 3-benzoyl-6,8-dimethylflavone, m. 191-2°.

IT 859805-98-4P, Chromone, 3-acetyl-2,6,8-trimethyl-, oxime
RL: PREP (Preparation)
(preparation of)

RN 859805-98-4 CAPLUS

CN Chromone, 3-acetyl-2,6,8-trimethyl-, oxime (3CI) (CA INDEX NAME)



L9 ANSWER 64 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1926:11317 CAPLUS Full-text

DOCUMENT NUMBER: 20:11317

ORIGINAL REFERENCE NO.: 20:1411g-i,1412a-h

TITLE: Action of hydroxylamine on chromones

AUTHOR(S): Wittig, Georg; Bangert, Fritz

SOURCE: Berichte der Deutschen Chemischen Gesellschaft [Abteilung] B: Abhandlungen (1925), 58B, 2636-42
CODEN: BDCBAD; ISSN: 0365-9488

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

GI For diagram(s), see printed CA Issue.

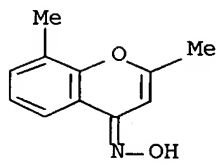
AB Although the C : O group of chromones reacts with extraordinary sluggishness with ketone reagents it shows a striking reactivity with NH2OH. From the observations of Harries on analogous compds. (Ann. 330, 190(1904)) it might be expected that NH2OH first adds at the double bond of the pyrone ring, with formation of a hydroxyaminochromanone (I) whose C:O group can now be oximated with ease, acidification of the product (II) splitting off NH2OH and yielding the chromone oxime (III). As a matter of fact, NH2OH in neutral solution with 2,8-dimethylchromone (IV) and 2,8-dimethyl-4-thiochromone gives the oxime (V) of IV. On the other hand, if IV is oximated in alkaline solution and the still warm reaction mixture is acidified with a dilute mineral acid, V is again obtained but if AcOH is cautiously added instead of the mineral acid there seps. a compound C11H14O8N2 (VI, R = 3,2-Me(HO)C6H2) which is also obtained from 6,2-Me(AcCH2CO)C6H3OH (VII) with NH2OH, showing that in the oximation of IV the pyrone ring is ruptured, with formation of the dioxime VI from which hot mineral acids hydrolyze the oxime group furthest from the C6H6 ring with formation of V. With cold acids VI gives, together with V, an alkali-soluble isomer (VIII). V and VIII cannot be converted into each other by concentrated alkalies or acids, and VIII, which, unlike V, gives a cornflower-blue color with FeCl3, can also be obtained from VI by the action

of alc. NH3, i. e., conditions under which chromone formation is impossible. VIII can therefore be only the hydroxyphenylisoxazole IX or X. It can also be obtained by heating VI at 160°. Since VI in general shows a tendency to split off the oxime group furthest from the C6H6 ring, IX is probably the correct formula for VIII. Alkaline oximation of 2,6-dimethylchromone (XI) yields an extremely unstable compound, apparently a hydroxyamino oxime (XII) in which the NHOH group is situated away from the C6H6 ring, as with cold acids it readily yields the oxime (XIII) of XI, also obtained with hot mineral acids without the formation of the hydroxyphenylisoxazole (XIV) (X, R = 5,2-Me(HO)C6H3), which can be obtained, together with XIII, by fusing the dioxime (XV) of 4,2-Me(AcCH2CO)C6H3OH (XVI). With alc. NH3, XV unexpectedly gave XII; apparently, in the absolute alc. NH3 added on the oxime group furthest from the C6H6 ring and on acidification was replaced by H2O; an analogous NH3 addition compound is probably an intermediate product in the formation of VIII from VI. On long heating with NaOH, XV gives an alkali-soluble compound C11H12O2N2 which by the Schotten-Baumann method yields a dibenzoate; apparently XV first forms the anhydride (cf. 2,6-dimethyl-3-acetochromone dioxime, preceding abstract) in which, under the further action of the alkali there occurs a shifting of the double bond with formation of the isoxdiazine XVII or XVIII (R = 5,2-Me(HO)C6H3); at the same time is formed an isomeric isoxdiazine which with Ac2O and NaOAc gives a diacetate and which doubtless also has 1 of the structures XVII or XVIII; the solubility in Na2CO3 of the 1st isomer and the slight solubility of the latter in NaOH indicate that they are XVIII and XVII, resp. 2-Acetylaceto-6-methylphenol dioxime (VI), obtained in 5.5 g. yield from 5 g. VII or in 75% yield from IV, m. 148-9° (slight decomposition), unchanged by heating with H2O under pressure. V, obtained almost quant. from VI in boiling aqueous alc. HCl, in 35% yield from the thiochromone with NH2OH in aqueous alc. and in about 75% yield from IV, m. 145.5-6.0°. α-[2-Hydroxy-3- methylphenyl]-γ-methylisoxazole (VIII), obtained in 80% yield from VI at 150-60°, in 30% yield from VI and alc. NH8, and in 0.3 g. yield, together with 0.4 g. V, from 1 g. VI in MeOH with cold 0.5 N HCl, m. 90.5-1.0°. 2-[α-Hydroximino-γ-hydroxamino-γ- hydroxyacetylaceto]-4-methylphenol (XII), m. 70-3°, loses H2O and solidifies and then has the m. p., 122-2.5°, of 2-acetylaceto-4-methylphenol dioxime (XV); both XII and XV give a blue color with alc. FeCl3. XIII, m. 184-5°. α-[2-Hydroxy-5- methylphenyl]-γ-methylisoxazole (XIV) (yield, 60%), m. 53-4°. 5-[2'-Hydroxy-5'-methylphenyl]3-methyl-1,2,6-isoxdiazine (XVIII) (3 g. from 4 g. XV refluxed 6-7 hrs. in excess of 2 N NaOH), m. 168-9° (slight decomposition), gives an olive-green color with FeCl3; dibenzoate m. 123.5-4.0°. 3,5-Isomer (XVIII) (yield, 0.4 g.), m. 185-7° (slight decomposition), gives no color with FeCl2, soluble in hot acids and alkalies and seps. unchanged on cooling; diacelate, m. 155.5-6.0°.

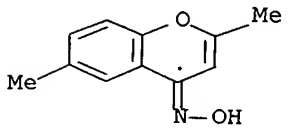
IT 56686-36-3P, Chromone, 2,8-dimethyl-, oxime 56686-37-4P, Chromone, 2,6-dimethyl-, oxime
RL: PREP (Preparation)
(preparation of)

RN 56686-36-3 CAPLUS

CN 4H-1-Benzopyran-4-one, 2,8-dimethyl-, oxime (9CI) (CA INDEX NAME)



RN 56686-37-4 CAPLUS
CN 4H-1-Benzopyran-4-one, 2,6-dimethyl-, oxime (9CI) (CA INDEX NAME)



L9 ANSWER 65 OF 66 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1920:19461 CAPLUS Full-text
DOCUMENT NUMBER: 14:19461
ORIGINAL REFERENCE NO.: 14:3633c-i,3634a-d
TITLE: Ring formation. I. Unsaturated ketones and chromanones from p-cresol
AUTHOR(S): v. Auwers, Karl; Lammerhirt, Elisabeth
CORPORATE SOURCE: Univ. Marburg
SOURCE: Ann. (1920), 421, 1-58
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 14:19461

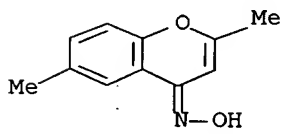
AB 3,6-Dimethylchromanone (C. A. 11, 2793) has been synthesized from p-MeC6H4OMe and Me2CBrCO2Cl in CS2 by the action of AlCl3, β-chloroisobutyro-p-cresol, yellow oil, b13 135-7°, being an intermediate product. 3-Bromo-3,6-dimethylchromanone, glistening prisms, m. 70-1°. Heated with PhNMe2 for 0.5 hr., this yields 3,6-dimethylchromone, glistening needles, m. 61-2°, b760 299-301°, b15, 165-8°. The structure of the chromone was established by synthesis from MeC6H3(OH)COEt and (CO2Et)2 by means of Na, and then heating with concentrated HCl, the 1st product being 3,6-dimethylchromone-2-carboxylic acid, needles, m. 234-6°; heated over the free flame, CO2 is evolved and the chromone formed. The reverse reaction is brought about by heating the chromone in EtONa for 0.5 hr. o-Propio-p-cresol p-nitrophenylhydrazone, orange-red, compact needles, m. 188-9°. 6-Methylchromone, from the Br deriv (C. A. 9, 84) by the action of PhNMe2, small, flat, glistening prisms, m. 88-9°. This was synthesized from HOC6H3MeCOMe and (CO2Et)2 by the action of Na, the intermediate product being ethyl 5-methyl-2-hydroxybenzoylpyroracemate, glistening, flat needles, m. 78-9°, which, upon hydrolysis, yielded 6-methylchromone-2-carboxylic acid, and this in turn the desired chromone. 2,6-Dimethylchromanone results by the action of MeC6H4OMe and MeCH:CHCOCl with 2 mols. AlCl3; glistening prisms, m. 54-5°, b10-5 138°, b20 152-3°, b26 162-3°, b760 262-3°. Semicarbazone, prisms, m. 203°. A by-product, separated by shaking the Et2O solution with NaOH, is 3,4-dimethyl-7-hydroxyhydrindone which seps. as the Na salt. By the use of 1 mol. AlCl3 there also results o-propenyl-p-cresyl ketone (o-crotonyl-p-cresol), golden transparent prisms and plates, m. 65-6°, b. 277-8°. MeCHClCH2CO2H, b20 110-3°, d420.2 1.1861, d419.85 1.1865, n 1.43992, 1.44213, 1.44828, 1.45327 for α, D, β and γ, at 19.85°, nD20 1.4421. β-Chlorobutyryl chloride, b21 51-3°, d420.06 1.2165, d420 1.217, n 1.44833, 1.45085, 1.45774, 1.46341 for α, D, β and γ at 20.05°, nD20 1.4511; gives with p-MeC6H4OMe β-chlorobutyro-p- cresol, b20 167-70°. Heated with dilute Na2CO3, this gives 2,6-dimethylchromanone. 3-Bromo-2,6-dimethylchromanone, glistening prisms, m. 104-5°. Heated with PhNMe2, this gives 2,6-dimethylchromone, compact needles, m. 102-3°. 5-Methyl-2-methoxybenzoylacetone, from MeOC6H3MeCOMe and AcOEt, yellow oil, b15 182-3°, d417.9 1.1196, d420 1.11, n 1.57582, 1.58562, 1.61562 for α, D and β at 17.9°, nD20 1.5847. Heated with HI, this gives 2,6-dimethylchromanone. p-Cresyl

crotonate, b27 153-5°, d420 1.059, nD20 1.5138, when treated with AlCl3 at 120° gave only 3,4-dimethyl-7-hydroxyhydrindone. The interaction of 1 mol. each of p-MeC6H4OMe, Me2C:CHCOCl and AlCl3 gives 2,2,6-trimethylchromanone, isolated as the semicarbazone, fine needles, m. 199-200°, and isobutenyl cresyl ketone, whose semicarbazone, m. 148-9°. With 2 mols. AlCl3 there results o-isobutenyl p-cresyl ketone, S-yellow prisms and long needles, m. 50°, b15 150-60°, d45.38 1.0376, nα 1.56280, nD 1.57178 With dilute NaOH it gives 2,2,6- trimethylchromanone; MeONa, boiling alc. HCl or H2SO4 or simple distillation causes the same change. It may be reduced to o-isovalero-p-cresol. Saturating a solution in AcOH with HCl gives o-[β-chloroisovalero]-p-cresol, compact prisms, m. 53-55°. o-[α,β-Dibromoisovalero]-p- cresol, pale yellow needles, m. 70-1°. A small amount of 3,3,4-trimethyl-7-hydroxyhydrindone, m. 67-8°, is also obtained in the above reaction, small compact prisms, m. 67-8°; semicarbazone, fine needles, m. 201-2°. 3,3-Dibromo-6-methylchromanone, compact, glistening prisms, m. 119-20°. Oxamino-oxime of 6-methylchromone, by the action of dilute alc. upon the chromone, prisms from MeOH, m. 143-4°. 3,6-Dimethylchromanone oxime, flat needles and prisms, m. 129-30°. p-Nitrophenylhydrazone, orange-red crystals, m. 179°. 3,6-Dimethylchromone oxime, prisms, m. 131-2°. 3,4,6-Trimethyl-4-chromanol, by the action of MeMgI upon dimethylchromanone, compact, flat prisms, m. 124°. 3,4,6-Trimethyl-α-chromene, prepared by the dehydration of the above alc. with P2O5, b18 135-6°. 2,6-Dimethylchromanone oxime, compact, glistening needles, m. 135°. Phenylhydrazone, compact prisms, m. 133°. p-Nitrophenylhydrazone, orange-red needles, m. 229-30°. 3,3-Dibromo-2,6-dimethylchromanone, compact, compact prisms, m. 100-1°. 2,6-Dimethylchromone oxime, fine needles, m. 151-2°. The Na salt is easily soluble in dilute NaOH. 2,4,6-Trimethyl-4-chromanol, small prisms, m. 89-90.5°. 2,4,6-Trimethyl-α-chromene, b25 138.5-9.5°. 2,2,6-Trimethylchromanone oxime, compact, glistening crystals, m. 130-1°. p-Nitrophenylhydrazone, orange-red glistening needles, m. 202°. 3-Bromo derivative. 3,3-Dibromo derivative, needles, m. 85°.

IT 56686-37-4P, Chromone, 2,6-dimethyl-, oxime
RL: PREP (Preparation)
(preparation of)

RN 56686-37-4 CAPLUS

CN 4H-1-Benzopyran-4-one, 2,6-dimethyl-, oxime (9CI) (CA INDEX NAME)



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ACCESSION NUMBER: 1908:7441 CAPLUS Full-text

DOCUMENT NUMBER: 2:7441

ORIGINAL REFERENCE NO.: 2:1709g-i,1710a-b

TITLE: Two Monohydroxy-α-Naphthoflavonols

AUTHOR(S): v. Kostanecki, St.

CORPORATE SOURCE: Univ. Lab., Bern

SOURCE: Berichte der Deutschen Chemischen Gesellschaft (1908), 41, 783-6
CODEN: BDCGAS; ISSN: 0365-9496

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

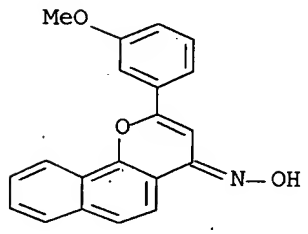
GI For diagram(s), see printed CA Issue.

AB 4'-Methoxy-α-naphthoflavanone (I below) from 2-anisalaceto-1- naphthol, HCl and MeOH. Colorless needles, m. 148°. Isonitroso derivative, yellow needles, m. and decomposes 169-70°. It gives orange colors with cobalt mordants and yellow ones with mordants of uranium, cadmium and lead. 4'-Methoxy-α-naphthoflavonol, by hydrolysis of the preceding compound with AcOH and dilute H2SO4. Slender, yellow needles, m. 249°. It gives light yellow colors with aluminum mordants and a light green, intensely fluorescent solution with concentrate H2SO4. Sodium salt, yellow and sparingly soluble. Acetyl derivative, colorless interlaced needles, m. 196°. 4'-Hydroxy-α-naphthoflavonol (II), from the methoxy compound and HI. Pale yellow plates, m. 293°. It gives light yellow colors with aluminum mordants. Concentrate H2SO4 dissolves it with a pale yellow color and an intense light green fluorescence; in aqueous NaOH the color is yellow with a greenish fluorescence. Diacetyl derivative, colorless needles, m. 181°. 3'-Methoxy-2-benzalaceto-1-naphthol, HOC10H6COCH:CHC6H4OMe, from m-methoxybenzaldehyde and 2-aceto-1-naphthol; orange-red needles, m. 115°. With concentrate H2SO4 the crystals darken and give a red solution. 3'-Methoxy-α-naphthoflavanone, from the preceding compound and HCl. Colorless needles, m. 130°. Isonitroso derivative, yellow crystalline powder, m. and decomposes 151°. It gives a pale yellow solution with dilute aqueous NaOH and orange colors with cobalt mordants. 3'-Methoxy-α-naphthoflavonol, yellow needles, m. 185°. In concentrate H2SO4, its solution is light yellow. With aluminum mordants it dyes pale yellow. Sodium salt, yellow and sparingly soluble. Acetyl derivative, colorless needles, m. 165°. 3'-Hydroxy-α-naphthoflavonol, from the methoxy derivative and HI. Lustrous pale yellow prismatic needles with 1EtOH, m. 248°. It dyes pale yellow with aluminum mordants. In concentrate H2SO4 the solution is pale yellow with a feeble greenish fluorescence. Sodium salt, slender yellow needles. In highly dilute solution it has a feeble greenish fluorescence.

IT 861550-15-4P, 7,8-Benzoflavanone, 3'-methoxy-, oxime
RL: PREP (Preparation)
(preparation of)

RN 861550-15-4 CAPLUS

CN 7,8-Benzoflavanone, 3'-methoxy-, oxime (1CI) (CA INDEX NAME)



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EAST Search History

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	8521	chromen$4	US-PGPUB; USPAT; FPRS; EPO; JPO; DERWENT	OR	ON	2007/07/11 19:10
L2	39180	oxime	US-PGPUB; USPAT; FPRS; EPO; JPO; DERWENT	OR	ON	2007/07/11 19:10
L3	2	chromen$4 adj (oxime)	US-PGPUB; USPAT; FPRS; EPO; JPO; DERWENT	OR	ON	2007/07/11 19:11
L4	8521	chromen$4	US-PGPUB; USPAT; FPRS; EPO; JPO; DERWENT	OR	ON	2007/07/11 19:11
L5	3	l4 and kinas	US-PGPUB; USPAT; FPRS; EPO; JPO; DERWENT	OR	ON	2007/07/11 19:11
L6	1645	l4 and kinase	US-PGPUB; USPAT; FPRS; EPO; JPO; DERWENT	OR	ON	2007/07/11 19:12
L7	654	l4 and (kinase adj inhibit$4)	US-PGPUB; USPAT; FPRS; EPO; JPO; DERWENT	OR	ON	2007/07/11 19:12
L8	195	l2 and l7	US-PGPUB; USPAT; FPRS; EPO; JPO; DERWENT	OR	ON	2007/07/11 19:12
L9	190	l4 and (protein adj kinase adj inhibit$4)	US-PGPUB; USPAT; FPRS; EPO; JPO; DERWENT	OR	ON	2007/07/11 19:13

EAST Search History

L12	970	546/114.ccls.	US-PGPUB; USPAT; FPRS; EPO; JPO; DERWENT	OR	ON	2007/07/11 19:16
L13	444	549/403.ccls.	US-PGPUB; USPAT; FPRS; EPO; JPO; DERWENT	OR	ON	2007/07/11 19:16
L14	878	544/279.ccls.	US-PGPUB; USPAT; FPRS; EPO; JPO; DERWENT	OR	ON	2007/07/11 19:16
L15	2247	l12 or l13 or l14	US-PGPUB; USPAT; FPRS; EPO; JPO; DERWENT	OR	ON	2007/07/11 19:17
L16	46	l15 and (kinase adj inhibition)	US-PGPUB; USPAT; FPRS; EPO; JPO; DERWENT	OR	ON	2007/07/11 19:17
S1	2	"20040198750"	US-PGPUB; USPAT; FPRS; EPO; JPO; DERWENT	OR	ON	2007/07/11 19:16
S2	2	("4065574").PN.	US-PGPUB; USPAT; FPRS; EPO; JPO; DERWENT	OR	ON	2007/07/11 17:28